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ELEMENTARY PRACTICAL CHEMISTRY

FOR USE IN THE LABORATORY

BY

P. W. OSCROFT, M.A. CANTAB.

AND

R. P. SHEA, M.A. OXON.

MASTERS AT UPPINGHAM SCHOOL

RIVINGTONS

34, *KING STREET, COVENT GARDEN*

LONDON

1910

PREFACE

THIS small hand-book is intended to indicate a course of Experiments in Practical Chemistry, which, taken in conjunction with a series of Lectures on similar lines, should give a boy a very fair knowledge of the subject.

Our reasons for placing yet another Practical Chemistry on a perhaps already overcrowded market are: firstly, that we think there is a certain novelty in the arrangement of the experiments, and also one or two novelties in the experiments themselves; and secondly, that we have omitted much matter usually included in such books, which we consider can be far more satisfactorily explained in a demonstration by the master.

Thus, we have intentionally not included any directions for the use of the Balance, and have also omitted any purely physical experiments, such as the determination of densities, and the proof of Boyle's Law, since these should, we think, have been already done in the course of Practical Physics which should always precede a Chemistry course.

We have included instructions for the Analysis of a Simple Salt, because our experience has shown that the intelligence of many boys is stimulated by this particular form of mental training, to a greater extent than is possible with any other course of School study.

It will be noticed that we are not enthusiastic advocates of the purely "heuristic" method of teaching Chemistry, as exemplified in many recent text-books; the time devoted to the subject is usually too short, and the pupil's intelligence unfortunately often too small, for the answering of the numerous conundrums which seem to be inseparable from that method.

We shall greatly appreciate any corrections or suggestions.

P. W. O.
R. P. S.

UPPINGHAM.

CONTENTS

CHAPTER I

PAGE

Chemical and Physical Changes 1

CHAPTER II

Experiments on Air 4

CHAPTER III

Experiments on Water 15

CHAPTER IV

Experiments on Chalk 30

CHAPTER V

Combustion and Flame 38

CHAPTER VI

The Halogens and their Acids 44

CHAPTER VII

Nitrogen and some of its Important Compounds . . 53

CHAPTER VIII

Sulphur—Its Various Forms, and some of its Important Compounds 58

CHAPTER IX

Compounds of Carbon 64

CHAPTER X

The Laws of Chemical Combination 67

CHAPTER XI

PAGE

Measurement of Volumes of Gases. Corrections for Temperature and Pressure 71

CHAPTER XII

Equivalent Weights 76

CHAPTER XIII

Acids, Bases, and Salts 83

CHAPTER XIV

Alkalimetry and Acidimetry 88

CHAPTER XV

Some Simple Volumetric Estimations 100

CHAPTER XVI

Analysis of a Simple Salt 112

Preliminary Examination for the Base 116

Confirmatory Tests for the Base 121

Preliminary Tests for Acids 126

Confirmatory Tests for Acids 127

Example Entry in Note-Book 130

Some of the more Common Gases 132

Table of Pressure of Aqueous Vapour 134

A MANUAL OF ELEMENTARY PRACTICAL CHEMISTRY

CHAPTER I

CHEMICAL AND PHYSICAL CHANGES

THOSE properties of matter which exist irrespective of its differences of composition, and those changes which affect the form and appearance of matter without altering its composition, are physical in their nature. Physical changes are, as a rule, of such a kind that their effects are not permanent, and are often brought about by affecting the cohesion of a substance.

Whenever a difference of composition is brought about, and the properties of the matter are dependent on its composition, there has taken place a change which is chemical in its nature.

Chemical changes result in the permanent alteration of substances, and the altered substances can only be restored to their original condition by further chemical change.

Experiment 1

Heat a piece of clean platinum wire or foil, holding it with the tongs in the flame of a Bunsen burner. Observe that it becomes very hot and glows red, but appears to undergo no other alteration. Take it out of the flame—in a minute it is cool enough to touch. Examine it closely,

and see if you can observe any burning, scorching, or tarnishing effect.

This change was purely Physical, not permanent in its effect.

Now take a strip of Magnesium wire or ribbon and treat it in the same way. The result is very different, for the metal takes fire almost at once, and burns with a brilliant light, giving off a white smoke and leaving a whitish, friable ash, which easily falls to powder.

Here the change is a Chemical one, and is permanent in its effect. We could not restore the metal to its former state without further chemical action.

Again, we may take a few pieces of Iodine and reduce them to powder in a mortar, but however finely they may now be divided, they are still Iodine, and will behave as Iodine, no matter to what reactions or chemical changes we may subject them.

Or if we heat the Iodine, it will be transformed into a vapour, which is quite different in appearance to the solid, but it still retains all the chemical characteristics of Iodine.

Changes such as these are purely Physical.

But, on the other hand, if we bring a small piece of Phosphorus into contact with the Iodine, a different result is obtained. Heat is produced, and a new substance, quite different in its properties to either Iodine or Phosphorus, will be formed. Here a Chemical change has taken place.

THE EFFECTS OF HEAT ON CERTAIN SUBSTANCES IN THE PRESENCE OF AIR

Experiment 2

To observe the effect of Heat on certain non-metals and metals.—Heat the following substances in air, using

the method indicated in each case. Heat gradually, and observe carefully.

Write in your notebook:—

(1) A description of the substance before it is heated.

(2) Your observations as to what happens during the heating.

(3) A description of the substance after it has been heated, and of any different substance which has been formed.

Carbon.—Hold a piece in the Bunsen flame, with the tongs.

Sulphur.—Heat some Flowers of Sulphur in a test-tube. Fill about half an inch, or an inch, if the bore is small, of the tube and heat cautiously at first, shaking the tube gently, or you will miss some of the changes.

Phosphorus.—Place some Phosphorus (a piece about the size of a small pea of the white or yellow Phosphorus, or as much as you can take up on the point of a penknife of the red variety) on a piece of broken porcelain. Hold the porcelain with the tongs in the flame.

Iodine.—Heat a few small pieces in a test-tube.

Aluminium (use foil).—Hold in the tongs in the flame.

Iron (wire).—Hold in the tongs in the flame.

Copper (wire or foil).—Hold in the tongs in the flame.

Zinc (foil).—Heat on porcelain.

Tin (foil).—Heat on porcelain.

Mercury.—Heat a little in a test-tube. Hold the tube in a clip.

Lead.—Heat in a small porcelain crucible without the lid. Support it on a tobacco-pipe triangle.

CHAPTER II

EXPERIMENTS ON AIR

Experiment 3

To find out whether the air has anything to do with the changes observed.—If we observe that the changes take place when the air is admitted, but not when it is excluded, we may fairly conclude that the air is in some way concerned in them.

(1) Place a few clean pieces of tin in a small porcelain crucible, and support it on a tobacco-pipe triangle. Now put some ordinary lard into an iron spoon, and warm it till it becomes liquid. Pour enough of the liquid lard into the crucible to cover the tin completely, thus shutting out the air.

Now heat the crucible, when the tin will melt. Continue heating, and observe that the surface of the melted tin remains bright, and does not tarnish as it did when the air was admitted. Record your observations.

(2) Take a strip of Magnesium ribbon about 20 cms. long, coil it up, and place it in a porcelain crucible, without the lid.

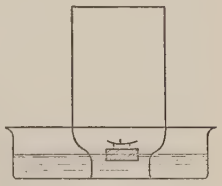
Weigh the crucible and Magnesium carefully. Now put on the lid, support the crucible as before, and heat strongly, raising the lid with the tongs every two or three minutes to admit the air, but being careful to let nothing escape. When all the Magnesium has been converted into ash,

allow the crucible to cool, remove the lid, and weigh again. There should be a decided increase in weight.

Thus we see that when the air was excluded there was no change, but that when the air was admitted there was a change both in appearance and weight—an increase.

Experiment 4

To find out if the whole of the air takes part in the change or not.—Place a piece of yellow Phosphorus the size of a small pea in a small porcelain dish placed on a large flat cork, floating in water in a pneumatic trough. Set the Phosphorus alight by touching it with a hot glass rod or wire, and at once cover it over with a glass bell-jar.

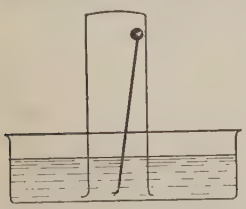


Record your observations and conclusions.

Part of the air seems to disappear. It combines chemically with the Phosphorus, producing a new substance, the white fumes or "smoke," which you observed in the bell-jar.

Experiment 5

To find out what proportion of the air combines with the Phosphorus.—Place an inverted gas-jar in a pneumatic trough containing water, and under it place a wire, having a small piece of Phosphorus fixed at the upper end. See the diagram. Leave it to stand till next lesson. Raise the jar from the bottom of the trough, but keep its mouth under water. Place a cover-glass over the mouth of the jar, and lift



it out on to the bench, mouth uppermost. Take off the cover, and insert a lighted taper. Pour the water in the jar into a graduated cylinder, and note its volume. Measure the volume of the whole jar, by measuring the water it holds when full. The volume of water in the jar when you first took it out of the trough represents the volume of air which has disappeared or combined with the Phosphorus. Calculate what fraction this is of the volume of the whole jar. It should be approximately one-fifth.

Experiment 6

To show that the air has something to do with the rusting of Iron.—Place three iron nails in a test-tube full of water which has been boiled to expel the air. Do the same with three more nails, but in addition pour a little melted vaseline into the tube, so as to form a layer above the water. Leave both tubes till next lesson. Record your observations.

Experiment 7

To show that there is an increase in weight when Iron rusts in air.—Cover a dry watch-glass with a thick layer of iron filings, and weigh the whole. Moisten the filings with a few drops of water, and leave them till next lesson. Heat the watch-glass on a sand-bath to drive off any moisture; allow it to cool, and weigh again. There should be an increase in weight.

Experiment 8

To find out what proportion of the air combines with the Iron when it rusts.—Take a gas-jar, and moisten the inside with water. Shake some iron filings into it, and holding the jar nearly horizontal, turn it round so that the

inside becomes coated with the filings. Stand the jar mouth downwards in water in a pneumatic trough, and leave it till next lesson. Slide a cover-glass over the mouth, and lift the jar on to the bench. Put a small quantity of Phosphorus in a deflagrating spoon, light it, and plunge it into the jar. The Phosphorus refuses to burn. Measure the volume of water in the jar, and also the whole volume of the jar, and make your calculation exactly as in Experiment 5.

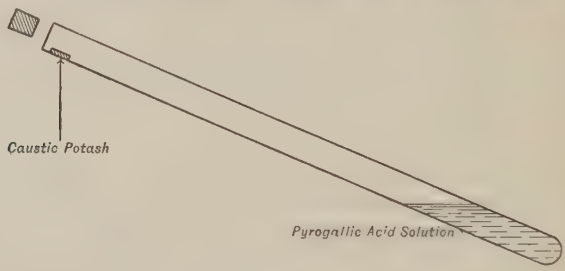
These experiments make it clear that air consists of two parts principally—an active part, which is concerned in the burning of Phosphorus or Magnesium, and in the tarnishing of lead, the rusting of iron, or the slow smouldering of Phosphorus—and an inactive part, which appears to have no effect on these substances, and in which a lighted taper or burning Phosphorus is extinguished.

The active part is called Oxygen, the inactive part is called Nitrogen. We have seen that about one-fifth of a given volume of air is Oxygen, the remaining four-fifths being the inactive Nitrogen.

Combustion, whether rapid, as in the case of burning Phosphorus, or a burning taper or piece of Magnesium wire, —or slow, as in the case of rusting iron or tarnishing lead, can only take place in air which contains Oxygen, and ceases as soon as the Oxygen is exhausted. Other substances besides Oxygen and Nitrogen exist in the ordinary air, but at present we are not concerned with these, and they only exist in comparatively very small quantities.

Experiment 9

To find the percentage of Oxygen in air.—Take a straight glass tube about 60 cms. long, and having a dia-



meter of about 3 cms., closed at one end and open at the other. Fit a good cork to the open end. Make a strong solution of Pyrogallic Acid in a glass beaker, and pour some into the tube, filling about 10 or 15 cms. of it. Now take a small piece of solid Caustic Potash, and place it just inside the open end of the tube, but do not allow it to slip down into the Pyrogallic solution. Cork up the open end of the tube, and tilt it so that the Caustic Potash slides down into the solution. This will cause the solution to turn brown, as it absorbs the Oxygen from the air in the tube. Hold the tube in a slanting position, so as to expose a large surface of the liquid to the air. When you consider that all action has ceased—say in five minutes—hold the tube in a vertical position, cork downwards, and mark the level of the brown solution on a gummed label stuck on the outside of

the tube. You can stick this label on while you are waiting for the action to cease, and at the same time stick on another about 10 cms. above it. This piece should be two or three inches long, as you do not know exactly where the second mark will be. See that the tube is dry when the labels are stuck on, or they will slip about and spoil your readings.

Now hold the corked end of the tube under water in a deep vessel, and take out the cork. The water will at once rise in the tube. Lower or raise the tube in the water till the levels are the same inside and out, and mark the level on the second label. Remove the tube from the water, and with a scale measure the distances AB, CB in the figure. Now calculate what per cent AC is of AB. Clearly AC represents the volume of Oxygen which was contained in the volume AB of air.

Whenever a substance combines with the Oxygen of the air, a new substance is produced. The white smoke of the burning Phosphorus, the ash of the burnt Magnesium, the reddish brown rust of the iron filings—these are all new substances, differing entirely in appearance and properties from those substances from which they were produced. Such substances are called Oxides—the substances formed by the combination of simple bodies with the Oxygen of the air.

We shall now see if we can split up one of these Oxides into its original components.

Experiment 10

To decompose the Oxide of Mercury into Oxygen and Mercury.—Place a small quantity of Red Mercuric Oxide (the ash or substance formed when Mercury is heated in air) in a test tube, and heat it in the Bunsen flame.

The red powder becomes darker in colour, and a bright deposit is formed on the sides of the tube near the mouth. This is Mercury, and small globules or drops of it may be scraped out on to a piece of paper. To recognize that Oxygen is given off, insert a glowing splinter of wood into the tube. This will at once burst into flame. If we continue the heating, practically the whole of the red powder will disappear, it being all decomposed or split up into Oxygen (gas) and Mercury (metal).

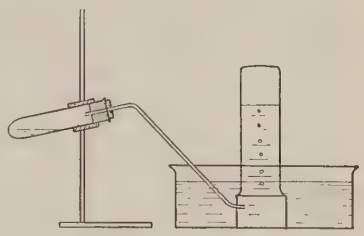
Experiment 11

To obtain Oxygen from Potassium Chlorate; to examine the substance left and compare it with the original.—Crush some crystals of Potassium Chlorate and heat them in a test-tube. The substance melts to a clear liquid, boils, and finally decomposes, giving off Oxygen, which may be tested with a glowing splinter as before. A white, solid substance is left clinging to the sides of the tube. Dissolve this out with a small quantity of water, place a few drops of the solution on a watch-glass, and allow it to evaporate. Examine the crystals with a magnifying glass, and notice that they are different from the original Potassium Chlorate crystals.

When we require Oxygen in any quantity—say four or five jars full—we usually obtain it by heating Potassium Chlorate. But we find that the gas is given off more regularly, and at a lower temperature, if we mix the Chlorate with the black powder known as Manganese Dioxide.

Experiment 12

To prepare Oxygen from Potassium Chlorate and black Manganese Dioxide.—Take a large test-tube or boiling-tube, and fill it about one-third full of a mixture of Potassium Chlorate (crushed in a mortar) and Manganese Dioxide, in the proportion of three to one. Fit a cork and delivery tube, and arrange the apparatus as in the diagram. Heat cautiously, beginning at the upper part of the tube, and remove the flame as soon as the gas begins to come off, applying it again when necessary. In this way fill six jars with the gas, and then remove the delivery tube from the water. Observe :—



(1) That the gas has no smell or colour.

(2) That a lighted taper or splinter plunged into it burns vigorously, but that the gas itself does not take fire.

(3) The effect of burning various substances in it.

Phosphorus.—A little red Phosphorus is ignited in a deflagrating spoon and plunged into a jar of the gas.

Sulphur.—In the same way.

Carbon.—Heat a small piece in a deflagrating spoon till it begins to glow red, and then plunge it into the gas.

Iron.—A strip of narrow, thin steel watch-spring is straightened out and fastened by one end to a deflagrating spoon. Bend the other end into a little hook, and take up on it a small quantity of Flowers of Sulphur. Set fire to this, and plunge the whole into the gas-jar.

N.B.—To avoid cracking the jar with the molten iron, put a layer of damp sand or a little water at the bottom.

Try to give names to the substances formed by these combustions in Oxygen.

Experiment 13

To find the percentage of Oxygen in Potassium Chlorate.—Weigh a hard glass tube, the size of a small test-tube, and closed at one end. Crush about half a gram of Potassium Chlorate, place it in the tube, and weigh again. Heat strongly till no more Oxygen comes off, holding the tube in a clip, and testing for Oxygen occasionally with a glowing splinter. Allow the tube to cool and weigh again.

Enter your results thus:—

	grms.
Weight of tube, empty	
„ „ and Chlorate . . .	
„ Chlorate taken	
„ tube and residue . . .	
Decrease of weight due to loss of Oxygen .	

Therefore grms. of Chlorate contain grms. of Oxygen.

Express your result as a percentage. It should be nearly 39 per cent.

Experiment 14

To find the weight of a litre of air.—(*a*) Take a dry *round*-bottomed flask of about 250 c.c. capacity. Fit it with a good rubber cork, having a short glass tube passing through it, with a short piece of rubber tubing and spring-clip attached. Weigh the flask, cork, tubes, and clip, with the clip open. You will have to support the flask on the balance with a short piece of thread. Now attach the tube to the air-pump, and exhaust the flask as far as possible. Close the clip and weigh again. There will, of course, be a decrease—the weight of the air which has been pumped out. But you will not have completely removed the air from the flask. To find out how much you have removed, open the clip under water. Lower the flask in the water till the levels are the same inside and out. Close the clip and take the flask out of the water. Now measure the volume of water in the flask by pouring it into a graduated cylinder. This is the same as the volume of air you pumped out. You know its weight, and can now find the weight of a litre of air.

Enter your results thus :—

	grms.
Weight of flask, etc., full of air . .	
„ „ after pumping .	
„ air pumped out . . .	
Volume of water entering flask . .	

Therefore c.c. of air weigh grms.

Hence find the weight of a litre.

(*b*) A second method, when a pump is not available.

Place about 50 c.c. of water in a *round*-bottomed flask fitted as before. Open the clip and boil the water, supporting

the flask over a wire gauze. When steam has been issuing steadily from the tube for about five minutes, close the clip and remove the flame at once. When cold, weigh the whole. Now open the clip, when the air rushes in. Weigh again. There will, of course, be an increase—the weight of the air which has entered. Measure the volume of water in the flask, and also measure the volume of the whole flask by filling it with water to the level of the bottom of the cork. Subtract the first volume from the second. This gives the volume of the air which entered the flask. You know its weight.

Make your calculation as before. Enter your results thus:—

	grms.
Weight of flask, etc., after air has been expelled	
„ „ „ opening clip . .	
„ air entering flask	
	c.c.
Volume of water left in flask	
„ whole flask	
Therefore volume of air entering flask . .	

Therefore c.c. of air weigh grms.

Hence find the weight of a litre.

At the temperature of an ordinary room your result should be near 1·26 grms.

CHAPTER III

EXPERIMENTS ON WATER

Experiment 15

To examine the purity of different kinds of water.—Take three or four drops of Distilled water, Rain-water, Tap-water, and Sea-water, each in a different watch-glass, place them on a sand-bath, and heat them gently till all the water has evaporated. Now examine the watch-glasses carefully, and describe what you see on each in your note-book.

Take a boiling-tube half full of tap-water, and heat it gently; notice that before it boils bubbles of gas are formed on the sides of the tube: this is air which was dissolved in the water.

Experiment 16

To find out whether a given substance is soluble or insoluble in water.—Use powdered Chalk, Plaster of Paris, and powdered Washing-soda. Take as much of each powder as would cover a sixpence, and place in three clean test-tubes; half fill each test-tube with distilled water, and shake vigorously with your thumb over the top. If the powder disappears, leaving a clear liquid, it is soluble in water; if not, it does not follow that it is insoluble: in that case proceed as follows:—

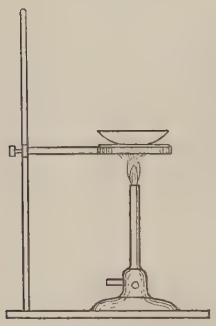
Filter three or four drops from each test-tube into differ-

ent watch-glasses, and evaporate to dryness, as in the last experiment.

Then, if there is a residue on the watch-glass, the substance is soluble in water; if there is no residue, it is insoluble.

Experiment 17

To determine the quantity of solid dissolved in one litre of Sea-water.—Clean and dry a porcelain dish and weigh it accurately; then by means of a pipette place in it 25 c.c. of sea-water. Boil this very gently on a wire gauze till it has nearly all boiled away and then add 25 c.c. more of the sea-water and continue boiling till all the water has disappeared. (N.B.—The last stages must be done over a very small flame, to avoid loss by spurting.)



When quite dry and cold, weigh again. The gain in weight of the dish will clearly be the weight of the solid dissolved in 50 c.c. of sea-water, and this weight multiplied by twenty will give the weight of solid in 1000 c.c., i.e. in one litre.

Enter your results thus:—

	grms.
Weight of dish, empty	
„ „ with solid at end of experiment	
Therefore weight of solid in 50 c.c. of sea-water	
„ „ „ 1000 c.c. . .	

Your result should be about 34 grms.

Experiment 18

To separate a mixture containing a soluble and an insoluble substance.—Make a mixture of sand and salt

and grind them together in a mortar; place a small quantity of the mixture in a boiling-tube and fill it half full of distilled water. Shake up well for a minute or two, to give the salt time to dissolve, and then filter the sandy mixture into a porcelain dish. Dry the filter-paper in its funnel over a sand-bath, and you will find on it the sand contained in the mixture. Evaporate to dryness the liquid which has come through the filter-paper into the dish, and you will get the salt which was in the mixture.

Experiment 19

To determine the percentage (*a*) of Saltpetre, (*b*) of Charcoal and Sulphur present in Gunpowder.—Gunpowder consists of an intimate mixture of Sulphur, Charcoal, and Saltpetre; of these the last is soluble in water, but the other two are not, so that they can be separated quite easily by the method of the last experiment.

Weigh out a clean, dry porcelain dish, a filter-paper, and 5 grms. of gunpowder, and proceed exactly as in the last experiment. Then if the dish and filter-paper are weighed, when quite dry, at the end of the experiment, the increase in weight of the dish will give the weight of the Saltpetre, and the increase in weight of the paper will give the weight of Charcoal and Sulphur in 5 grms. of gunpowder.

Enter your results thus:—

	grms.
Weight of porcelain dish, empty . . .	
„ „ „ and Saltpetre .	
Therefore weight of Saltpetre . . .	
Weight of filter-paper	
„ „ with Charcoal and Sulphur	
Therefore weight of Charcoal and Sulphur .	

Therefore 5 grms. of gunpowder contain grms. of Saltpetre and grms. of Charcoal and Sulphur.

Hence calculate the weights in 100 grms. of gunpowder.

Gunpowder usually contains 75 per cent. of Saltpetre.

Experiment 20

To determine the number of grms. of Salt which can be dissolved by 100 grms. of water at the temperature of the laboratory.—Prepare a solution of Salt by taking a beaker half full of distilled water, and stirring up Salt in it till no more will dissolve, though there is still some Salt left at the bottom of the liquid; this solution is called a "saturated solution" of Salt. Allow the Salt to settle, and pour off about 5 c.c. of the clear liquid into a clean porcelain crucible which you have previously weighed; then weigh it again with the liquid in it, and then place it in a drying oven, and, keeping the temperature at about 120° C., evaporate off all the water till it is quite dry. When your crucible is quite cold, weigh it again. Then the difference between the second and third weights will be the weight of the water driven off, and the difference between the first and third weights will be the weight of the Salt which was dissolved in it.

Enter your results thus:—

	grms.
Weight of crucible, empty	
„ „ and Salt solution . .	
„ „ after heating . . .	
Therefore weight of water evaporated . .	
And weight of Salt dissolved in it . . .	

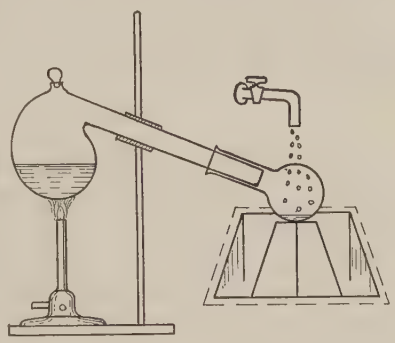
Therefore grms. of water dissolve grms. of Salt.

Hence calculate the weight of Salt which 100 grms. of water will dissolve.

An accurate experiment will give between 35 and 36 grms.

Experiment 21

To prepare some distilled water.—Make some tap-water impure and turbid by dissolving in it a little powdered Copper Sulphate, and also suspending in it a little powdered chalk. Place this liquid in a retort connected with a clean flask, as in the diagram, and arrange it so that cold water from a tap either drips on to the flask or can be poured over it. Then boil the water; the steam, which contains none of the impurities in the water, is condensed in the flask, and if a drop or two of this "distilled" water be evaporated on a watch-glass it should leave no residue.



Experiment 22

To prepare crystals of various substances.—Take some powdered Saltpetre and prepare a saturated solution of it in water in a boiling-tube, as in Experiment 20.

Now warm this solution gently, when you will find that the Saltpetre which was left at the bottom of the tube now dissolves, which proves that warm water will dissolve more of it than cold water.

If you allow this warm solution to cool slowly, the cold

water can no longer hold the Saltpetre in solution, and so deposits it in the form of crystals, which, if you notice them carefully, you will see to be all of exactly the same shape.

Prepare also in the same way crystals of Alum and Copper Sulphate. Notice that the crystals of each substance are like one another, but different from those of other substances.

Draw diagrams in your notebook, showing the shape of all these sets of crystals.

Experiment 23

To examine the effect of Heat upon different kinds of crystals.—Take a small quantity of each of the crystalline substances prepared in Experiment 22, dry it, and heat separately in small dry test-tubes. Notice that in two cases steam is driven off from the crystals, but in the other case the substance melts, but does not give off any steam.

This water, which is found in some kinds of crystals, is called "water of crystallization."

Take a little of the white powder which is left after heating the Copper Sulphate crystals (it is called "Anhydrous" Copper Sulphate), and add one or two drops of water; notice that the powder becomes quite hot, and also that it recovers its blue colour again. This powder is a very good test for the presence of water.

Heat also a few crystals of Ammonium Chloride (Sal-Ammoniac) and Iodine in small test-tubes; notice that they change at once into vapours which are deposited again as small crystals on the cool part of the tube. This process is called "Sublimation."

Experiment 24

To determine the percentage of Water contained in crystals of Copper Sulphate.—Clean, dry, and weigh a small porcelain crucible and its lid; weigh into it about half a gram of powdered crystals of Copper Sulphate. Heat the crucible with its lid on gently over a very small flame for about ten minutes. On no account must it be allowed to get red-hot. Allow it to cool, and weigh again, repeating this heating and cooling and weighing till no more weight is lost.

Then clearly the loss of weight is that of the water contained in the crystals of Copper Sulphate.

Enter your results thus :—

	grms.
Weight of crucible (with lid), empty . .	
„ „ „ and crystals .	
Therefore weight of crystals taken . . .	
Weight of crucible, lid, and Copper Sulphate after heating	
Therefore weight of water of crystallization .	

Therefore grms. of Copper Sulphate contain grms. of water.

Hence calculate the weight of water contained in 100 grms. of Copper Sulphate

A good result should be near 36 per cent.

Experiment 25

To investigate the action of Soap on water.—Cut up some shavings of ordinary yellow soap, and warm them in a boiling-tube nearly full of distilled water till they have all dissolved.

While you are doing this, put some tap-water in a beaker, boil it for five minutes, and set it aside to cool. Now take 25 c.c. of distilled water in a glass-stoppered bottle, add to it a little of the soap solution, put in the stopper, and shake vigorously; if no lather forms, keep on adding the soap solution in small quantities, noticing roughly how much of it must be added to produce a lather which lasts one minute.

Now do the same experiment with 25 c.c. of ordinary tap-water, and you will find that much more soap solution has to be added to it before a permanent lather is obtained.

A water, like distilled, which lathers readily with soap is called "soft" water, while one, like tap-water, which requires a lot of soap to form a lather, is called "hard" water.

Now take 25 c.c. of the boiled tap-water, and add the soap solution to it till a lather is formed: you will find that you do not need so much soap solution as for the unboiled tap-water. This shows that boiled tap-water is "softer" than ordinary tap-water.

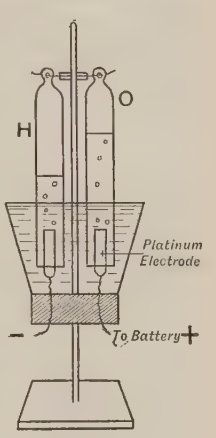
The "hardness" of water is due to the impurities of Chalk and Sulphate of Calcium, which are dissolved in the water.

Experiment 26

To analyse water by Electrolysis.—Arrange an apparatus for the Electrolysis of water as in the diagram, putting water acidulated with a few drops of Sulphuric Acid in the basin and in the test-tubes, and using two accumulators or Bichromate cells for the electric current.

The water is split up into two gases, which collect in the

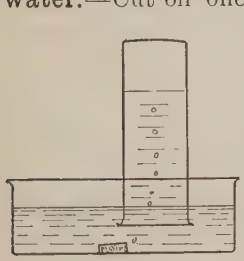
test-tubes; notice that the one connected with the Negative terminal of the battery gives off gas twice as fast as the one joined to the Positive terminal. When a fair amount of the gases is collected, test the smaller quantity with a glowing splinter of wood: it is rekindled, and therefore the gas must be Oxygen. If a lighted taper be applied to the gas in the other tube, it will be found to burn with a blue flame and a small explosion: this gas is called Hydrogen.



By this experiment we have proved that water consists of the two gases Oxygen and Hydrogen, in the proportion of two volumes of Hydrogen to one of Oxygen.

Experiment 27

To investigate the action of the metal Sodium on water.—Cut off one or two pieces of Sodium about the size of half a pea (N.B.—the fingers and knife must be perfectly dry), and put them in a short piece of lead piping closed up at one end, and then half close up the other end of the pipe.



Place the lead pipe with the Sodium inside it at the bottom of a shallow basin of water; bubbles of gas are given off, which may be collected in a test-tube full of water held over the end of the pipe.

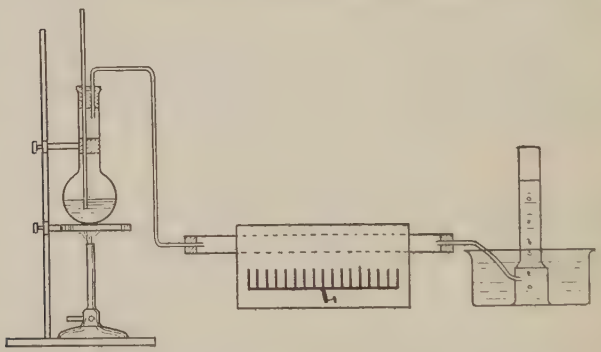
Test the gas in the tube with a lighted taper; it burns with a blue flame and a small explosion, and is therefore Hydrogen.

Now test the water in the basin with red litmus paper: it is turned blue; this is because of the Caustic Soda (Sodium Hydroxide) which has been formed in the water. We have found, then, that when Sodium acts upon water Hydrogen gas is given off and Sodium Hydroxide is formed.

$$Na + H_2O = NaOH + H.$$

Experiment 28

To find out what happens when steam is passed over red-hot iron.—Take an iron tube about 15 in. long,



put some iron filings into it, fit good corks with delivery tubes in to each end of the tube, and then place it in a furnace. When thoroughly red-hot, pass a slow current of steam through one of the delivery tubes and connect up the other to a pneumatic trough for collecting any gas which may come off (see diagram).

A gas is found to collect in the jar, which burns with a slight explosion if a light is applied; this gas is Hydrogen.

When the apparatus is cool, take the iron filings out of

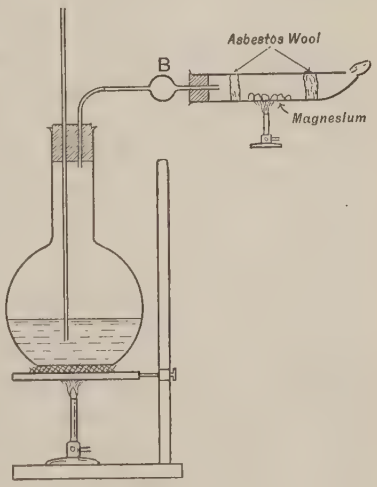
the tube and examine them carefully. They will be found to be covered with a black coating, which is the Magnetic Oxide of Iron.

Steam and iron, therefore, produce Hydrogen and Magnetic Oxide of Iron.

$$3Fe + 4H_2O = 4H_2 + Fe_3O_4.$$

Experiment 29

To examine the action of heated Magnesium on steam.—Take a small flask with a little water in it, also a cork to fit it having two holes bored through it: through one of the holes place a long straight piece of glass tubing, open at both ends, to act as a safety-valve, and through the other hole fit a short piece of glass tubing bent at right angles. Next get a test-tube, and by melting the closed end in the flame, blow a small hole through it. Put inside the test-tube a coil of Magnesium wire, and by means of a cork fit it on to the bent piece of tubing, as in the diagram. Boil the water in the flask gently, and as soon as the steam comes off, heat the Magnesium strongly: it will soon take fire and burn vigorously in the steam: Hydrogen gas comes off and burns as a jet at the hole in the test-



tube, a white powder, Magnesium Oxide, being left in the tube.

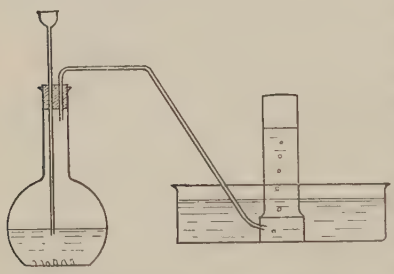
The Magnesium has taken Oxygen from the steam, using it in burning, and has liberated the Hydrogen.

$$Mg + H_2O = MgO + H_2.$$

The small bulb shown at B in the figure is useful to prevent water being carried into the test-tube, but is not essential. It may also be as well to put a little wad of Asbestos wool on each side of the Magnesium in the test-tube.

Experiment 30

To prepare Hydrogen, and to examine some of its Properties.—Fit up a flask—or, better, a Wolff's bottle—with a thistle funnel and delivery tube; pass the tube into a pneumatic trough full of water and provided with a bee-hive and gas-jars, as in the diagram. Put some granulated Zinc in the flask, cover it with an inch of water, and add a little strong Sulphuric acid or Hydrochloric acid through the thistle funnel: Hydrogen gas is evolved and is collected in the gas-jars. Obtain several jars of the gas, and observe carefully its properties, doing the following experiments to prove them:—



(1) Note that it is colourless and has no smell.

(2) Prove that it is lighter than air by pouring it upwards from one jar to another containing air.

(3) Show that it does not dissolve in water, by shaking up a little water in a jar of it, and noting that the glass plate does not stick to the mouth of the jar when it is held upside down.

(4) Put a lighted taper to the mouth of the jar, held mouth downwards. Notice that the Hydrogen burns with an explosion. Now push the taper right into the jar, and notice that it is put out, showing that Hydrogen is not a supporter of combustion.

Now take some of the liquid left in the flask in a porcelain dish, boil it down to one-quarter of its original bulk, and allow it to cool. Crystals are formed in the dish: these are called Zinc Sulphate.

We see, then, that when dilute Sulphuric acid acts on Zinc, Hydrogen gas is liberated and Zinc Sulphate is formed.

N.B.—If Hydrochloric acid is used instead of Sulphuric acid, the salt formed is called Zinc Chloride, and this will not form crystals on evaporation.

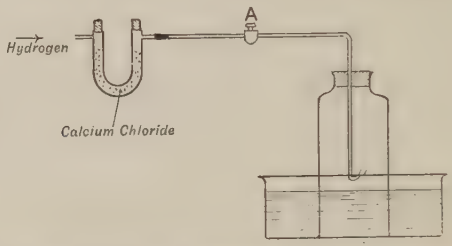
$$Zn + H_2SO_4 = ZnSO_4 + H_2.$$

Experiment 31

To show that when Hydrogen burns in air it is uniting with the Oxygen.—Use the apparatus for preparing Hydrogen as in Experiment 30. Pass the gas first through a **U**-tube containing Calcium Chloride to dry it, then through a stop-cock (A), and burn it at a jet, the tube of which passes through a large rubber cork. Insert the cork in a large bell-jar full of air, standing in a trough containing water as in the figure. Watch the flame of burning Hydrogen carefully, and the moment it goes out close the stop-

cock (A), and at once disconnect the Hydrogen apparatus to prevent the acid being forced back through the thistle funnel.

When the bell-jar is cool you will notice that the water



has risen one-fifth of the way up the jar, and, if tested, the gas left in the jar is found to be Nitrogen. The Hydrogen, therefore, must have combined with the Oxygen of the air when burning.

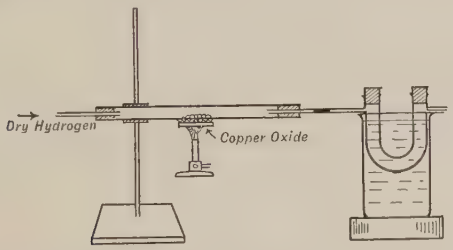
Experiment 32

To show that when Hydrogen burns in air water is formed.—Use the apparatus for preparing dry Hydrogen as in Experiment 31, but burn the gas at a straight jet, and allow the flame to play on a boiling-tube full of cold water. After a time drops of liquid are formed on the outside of the tube and will drop off into a watch-glass placed underneath. If now you add Anhydrous Copper Sulphate (see Experiment 23) to this liquid, it is found to be turned blue by it, proving that the liquid formed when Hydrogen burns in air is water.

$$H_2 + O = H_2O.$$

Experiment 33

To show that Hydrogen will take Oxygen from a metallic Oxide and form water.—Place some black Copper Oxide in a piece of hard glass tubing about nine inches long; connect the tube at one end with an apparatus for supplying dry Hydrogen gas, and at the other with a clean empty U-tube kept cool by being immersed in a beaker full of cold water. After the Hydrogen has been passing through the apparatus for some time, heat the tube containing the Copper Oxide for five minutes. When cool, examine its contents, which will be found to consist of pure Copper,



so that Oxygen must have been taken away from them. At the same time a liquid has been collected in the cooled U-tube which, if tested with Anhydrous Copper Sulphate, is found to be water.

The Hydrogen has taken Oxygen from the Copper Oxide to form water, which can therefore consist of nothing but Oxygen and Hydrogen.

Such an action as this is called a "Reduction," and the Hydrogen is called a "Reducing Agent."

$$CuO + H_2 = Cu + H_2O.$$

CHAPTER IV

EXPERIMENTS ON CHALK

Experiment 34

To investigate the effect of heat on Chalk.—Take one or two grams of powdered or precipitated Chalk in a porcelain crucible, place the crucible on a pipe-clay triangle, and heat very strongly in a Bunsen flame, or, if possible, with a blow-pipe, for ten or fifteen minutes.

The residue left after heating consists mostly of Lime, and possesses quite different properties from Chalk. Add a few drops of water to the Lime, and also to some of the original Chalk; notice that the Lime combines with the water and becomes quite hot, but that nothing happens in the case of the Chalk.

Test the solubility of both Lime and Chalk in water (see Experiment 16); notice that Lime is soluble but Chalk is insoluble in water.

$$CaCO_3 = CaO + CO_2.$$

Experiment 35

To find the proportion of Lime present in Chalk.—Weigh accurately a clean platinum or porcelain crucible, put in it about half a gram of precipitated Chalk, and weigh again. Heat the crucible and its contents strongly for twenty minutes in a blow-pipe flame, or a small furnace if available; when cool, weigh again. After weighing heat

again for five minutes, allow to cool, and weigh once more. The weight should be exactly the same as the previous one; if not, it must be again heated, cooled, and weighed till the weight no longer decreases.

Then the residue in the crucible is all Lime, and from its weight the proportion of Lime to chalk can be calculated.

Enter your results thus:—

	grms.
Weight of crucible, empty . . .	
„ „ and Chalk . . .	
Therefore weight of Chalk . . .	
Final weight of crucible and Lime . .	
Therefore weight of Lime	

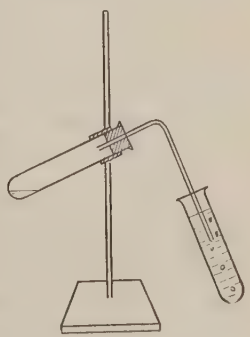
Therefore grms. of Chalk contain grms. of Lime.

Hence calculate the weight of Lime in 100 grms. of Chalk.

A good result gives 56 per cent. of Lime.

Experiment 36

To prove the presence of Carbonic Acid gas in Chalk.—Place a little chalk in a small test-tube made of hard glass; fit it with a cork and delivery tube as in the diagram, allowing the delivery tube to dip a little way only under the surface of some Lime-water placed in another test-tube. Heat the Chalk very strongly with a Bunsen burner or a blow-pipe: bubbles of gas come out of the delivery tube, and in a short time the Lime-water in the second test-tube



turns milky. Now the only gas which turns Lime-water milky is Carbonic Acid gas, which must therefore be coming off from the Chalk. By this experiment we have proved that Chalk contains Carbonic Acid gas, and the last experiment showed that it contained Lime.

$$CaCO_3 = CaO + CO_2.$$

Experiment 37

To investigate the effect of acids on the Carbonates of different Metals.—Take small quantities of each of the Carbonates of Calcium, Sodium, and Copper in different test-tubes, and add to each a little dilute Hydrochloric acid. Also to small quantities of the same Carbonates add dilute Sulphuric and Nitric acids.

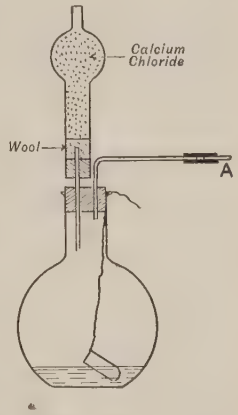
Note that in every case a gas is evolved, and if a drop of Lime-water is held on a glass rod in the mouth of the test-tube, it is in every case turned milky, proving that the gas evolved is Carbonic acid gas. Evaporate a little of the clear liquid left in each test-tube, and examine the solids left behind. Describe each briefly, noting particularly whether they are crystalline or amorphous.

In the case of Hydrochloric acid, the salt left behind is the Chloride of the metal; with Sulphuric acid it is the Sulphate, and with Nitric acid it is the Nitrate of the metal which is produced.

Experiment 38

To find the weight of Carbon Dioxide evolved on treating a known weight of Calcium Carbonate with an acid.—Fit up the apparatus shown in the diagram, taking care that all joints are gas-tight. Pour dilute Hydrochloric acid into the flask. Weigh carefully a small test-tube;

introduce about 1 grm. of precipitated Chalk, and weigh again. Support the tube and Chalk by a thread, as in the diagram. Weigh the whole apparatus.



Now tilt the flask so that the acid comes in contact with the Chalk. Repeat this till there is no further action, i.e. until no more solid remains.

Warm the flask very gently to expel any gas dissolved in the water.

The apparatus contained air when you weighed it, and now contains Carbon Dioxide. Suck this out through the drying-tube, opening A at the same time. Close A, and re-weigh the whole. Enter your results thus :—

	Grms.
Weight of small tube	
„ „ „ and Chalk . . .	
„ Chalk used	
„ whole apparatus before action .	
„ „ „ after „ .	
„ Carbon Dioxide driven off . .	

From these observations calculate the percentage of Carbon Dioxide in Calcium Carbonate.

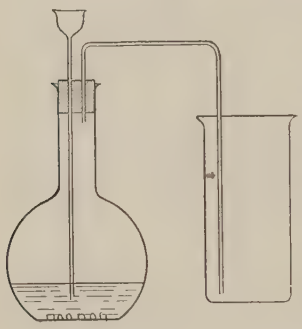
The same determination may be made as follows :—

Weigh a small porcelain crucible with its lid. Introduce about a gram of dry powdered Marble, and weigh again. Now heat the crucible very strongly, without the lid. This is best done in a small muffle furnace, but it may also be conveniently done over a foot blow-pipe. When the heating is finished, replace the lid, and allow the crucible to cool

in a desiccator. When cold, weigh again. The loss of weight represents the Carbon Dioxide which has been driven off. Calculate the percentage as before. A good result would be near 44 per cent.

Experiment 39

To prepare Carbonic Acid gas, and examine some of its properties.—Arrange a flask as in the diagram, with thistle funnel and delivery tube for collecting the gas evolved by downward displacement. Place in the flask some broken pieces of Marble, and add dilute Hydrochloric acid slowly down the funnel; Carbonic Acid gas comes off briskly; collect four or five jars of it, and perform the following experiments with it:—



(1) Place a lighted taper in one jar and notice that it is at once put out, showing that the gas does not support combustion. Observe also that the gas does not burn itself.

(2) Shake up a little water in another jar of the gas, holding a glass plate firmly over the mouth; notice that the plate sticks to the jar, showing that a partial vacuum has been formed inside owing to the solubility of the gas in water.

(3) Place a short piece of lighted candle on the bench and extinguish it by pouring the gas from another jar over it; this shows that the gas is heavier than air.

(4) Place a spoonful of Petrol or Turpentine in an iron dish, light it, and pour over it the contents of a large jar of the gas: notice how readily the gas acts as an extinguisher.

(5) Take some blue litmus solution in a test-tube, and bubble some of the gas through it; note that the colour is changed to a pale claret colour, not to a true acid red, because Carbonic Acid is a weak acid. Now boil the litmus solution, and as the gas is boiled off it will regain its original blue colour.

Try the effect of boiling some litmus solution reddened with Hydrochloric Acid, and show that the red colour is permanent.

(6) Take a strip of Magnesium ribbon about four inches long in the tongs, light it, and lower it into a jar of the gas; note that it continues to burn with a spluttering noise; examine very carefully the Magnesium Oxide which is left; there will be seen on it some black specks: these consist of Carbon derived from the Carbonic Acid gas.

(7) Bubble some of the gas through Lime-water contained in a test-tube; notice that the Lime-water is first turned milky, but on continuing to pass the gas it becomes clear again; the milkiness is due to the formation of Chalk which is insoluble in water, but is dissolved by the excess of Carbonic Acid gas. Now boil the clear liquid, and as the gas is driven off, notice that Chalk is again precipitated.

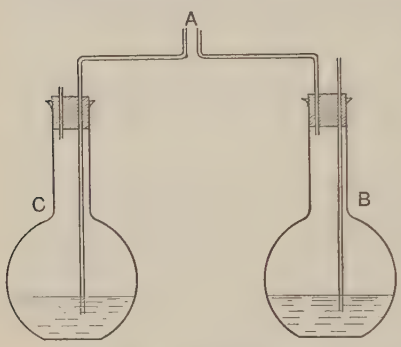
When all these experiments are done, pour off some of the clear liquid from the Marble in the flask into a porcelain dish and evaporate it to dryness.

The white flaky solid left behind is called Calcium Chloride, and if left exposed to the air for an hour or so, it absorbs moisture and becomes a sloppy mass; substances which do this are called "deliquescent."

$$CaCO_3 + 2HCl = CaCl_2 + CO_2 + H_2O.$$

Experiment 40

To prove the presence of Carbonic Acid gas in air and in air breathed out from the lungs.—Leave a few cc. of Lime-water exposed to the air in a watch-glass for half an hour; notice that it becomes milky, showing that there must be Carbonic Acid gas in the air. Arrange two small flasks containing Lime-water with tubes as in the figure, so that, if the mouth be applied at A, air can be drawn in through the flask B and breathed out again through the flask C. After two or three breaths have been taken, note that the Lime-water in C has turned milky, while that in B is still quite clear; this shows that breathed-out air contains more Carbonic Acid gas than ordinary air, and that this gas is therefore formed inside the body.



Experiment 41

To prepare crystals of washing Soda.—Set up the apparatus for preparing Carbonic Acid gas and bubble the gas, as it comes off, through a boiling-tube containing about 100 cc. of a strong solution of Caustic Soda (Sodium Hydrate). Notice that at first the bubbles of gas disappear before they reach the surface, showing that they are absorbed by the Caustic Soda; notice also that the liquid becomes warm, showing that chemical combination is taking place between the Caustic Soda and the Carbonic Acid gas.

After about ten minutes' steady bubbling, put the liquid from the boiling-tube into a porcelain dish and boil about half of it away, then, on allowing it to cool, crystals should be formed at the bottom of the dish. Examine these carefully; they are crystals of Washing Soda, or Sodium Carbonate. Dry them well, and leave them in a watch-glass exposed to the air for a few days; notice that a white powder is formed on the surface: this is because the crystals have given up some of their water of crystallization, and have become Anhydrous. Crystals which do this are said to be "efflorescent."

CHAPTER V

COMBUSTION AND FLAME

CERTAIN substances, such as Paraffin oil, Alcohol, Sulphur, and Phosphorus, when heated in air begin to combine so rapidly with the Oxygen in the air that they burst into flame; such substances are said to be "combustible," and the air is said to be a "supporter of combustion."

The temperature at which the flame forms is always the same for the same substance, and is called its "temperature of ignition."

Experiment 42

To find out the different temperatures at which substances begin to burn.—(1) Take a very small piece of Phosphorus (yellow) on a broken piece of porcelain, heat a glass rod till it is just uncomfortably warm to the hand, and touch the Phosphorus with it. The temperature at which this ignition takes place is below 50° C., and therefore Phosphorus must be very carefully handled.

(2) Take 2 or 3 cc. of Carbon Disulphide in a porcelain dish, use the same glass rod as in the last experiment, heat it in the flame, and dip it into the liquid from time to time as it gets hot; notice that, long before it gets red-hot, it sets fire to the Carbon Disulphide, which is a very inflammable substance.

(3) Do the same experiment with Alcohol; notice that its temperature of Ignition is very much higher.

(4) Take about 25 cc. of ordinary Paraffin oil in a porcelain dish, bring a lighted taper near the surface of the oil; notice that it does not catch fire. Now warm it very slowly over a very small gas flame, holding a thermometer in the liquid; test it from time to time by bringing a small flame near the surface; at a certain temperature (note it) it will take fire. This temperature is called the "Flash-point" of the Paraffin.

(5) Do the same experiment with Colza oil, and notice that its "flash-point" is very much higher.

(6) Hold a glowing splinter of wood to coal-gas as it issues from a jet, and notice that its temperature is quite high enough to ignite the gas.

(7) Press down a square of wire gauze over a lighted Bunsen flame; notice that the flame does not pass the gauze, though the gas itself has come through, for it may be lighted by a taper; if, however, the gauze be kept down till it is red-hot, it will ignite the gas above it.

Next hold the same piece of wire gauze over an unlighted burner, and light the gas above it with a taper. The flame will not penetrate below the gauze, because the metallic wire of which the gauze is made conducts the heat away, so that the under side of the gauze always remains below the ignition temperature of the gas. A flame entirely surrounded by fine wire gauze is used by miners to prevent their lamps igniting the "fire-damp" in a coal mine; it was invented by Sir Humphry Davy and is called the Davy lamp.

Experiment 43

To study a candle flame.—Cut a lump of wax from a candle and hold it in a Bunsen flame. Notice that it melts but does not catch fire: solid wax, therefore, does not burn.

Now put the lump of wax in a porcelain crucible, and melt it; keep applying a lighted taper to the liquid wax from time to time as its temperature gets higher; notice that it is not till the wax boils and has become a gas or vapour that it will take fire.

Next set up the candle on the bench; light it and examine carefully the structure of the flame; make a sketch of it in your notebook.

It consists of three parts or zones:—

(1) A bluish transparent inner zone immediately surrounding the wick. This consists of unburnt gaseous wax rising from the wick.

(2) A yellowish white middle zone surrounding the inner zone. This gives out all the light of the candle, and is called the zone of "incomplete combustion."

(3) An almost invisible outer zone enveloping the whole flame. This is called the zone of "complete combustion."

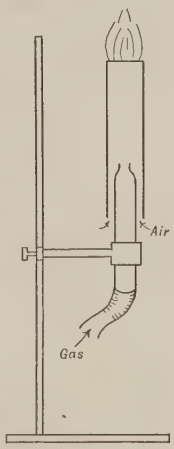
To show that the candle flame is hollow and cool in the middle, lower a piece of paper on to the flame till it is just above the wick; take it away before it catches fire, and notice that a brown ring has been scorched on the paper, leaving the part over the centre of the flame unscorched. To show that the inner zone consists of unburnt gaseous wax, heat a piece of thin glass tubing, about three inches long, in a Bunsen flame, and then hold one end of it in the inner zone, with the other end inclining upwards; notice that unburnt gas rises up the tube and may be lighted at the top. Hold

a porcelain dish in the candle flame; notice that a black deposit is formed on the dish. This consists of Carbon particles, which being set free and raised to a white heat in the middle zone, gave out all the light: they are eventually burnt in the outer zone, but if not, the flame smokes.

Take a mouth blow-pipe, and blow gently through the flame with it: notice that the whole of the light disappears, and only a blue flame is produced. This is because the extra Oxygen supplied by the blow-pipe has caused the Carbon to be burnt at once, and so no particles are set free and raised to a white heat. It also concentrates the flame and makes it hotter.

Experiment 44

To study a Coal-gas flame.—Take a piece of glass tubing of 4 mm. bore and about 5 in. long; soften it in the centre of a Bunsen flame, and pull it out till it forms two jets.



Take one of these, and join it up to the gas supply, and light the gas as it issues from the jet.

Examine the structure of the flame carefully, note any point in which it resembles or differs from that of a candle, and do all the experiments with it that you did with the candle flame. Notice that it behaves in exactly the same way in every case.

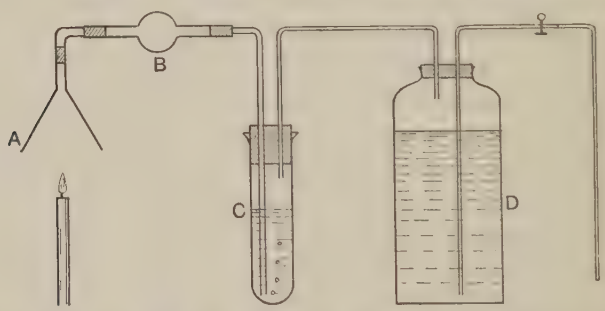
Now fix the jet in the clip of a retort-stand, and place over it a piece of wider glass tubing about 5 in. long, fixed in another clip, as in the diagram. Turn on the gas, and light it as it issues from the top of the upper tube. Notice that the gas now burns with a blue, non-luminous flame:

this is because air is now drawn in with the gas at the bottom of the wider tube and supplies sufficient Oxygen to burn all the Carbon at once, so that no particles are set free. This flame is called the Bunsen flame, after its inventor. It is hotter than the ordinary gas flame, because the area of combustion is smaller and the heat is concentrated. By raising the wide tube higher, the flame becomes smaller still, as more air is admitted, and the gas burns with a roaring noise. This flame is very hot indeed.

Examine the flame carefully; note that it consists of two zones only—an inner pale blue zone, which is quite cool (it may be proved so by the same experiments as for the candle flame), and an outer purple zone which is very hot indeed.

Experiment 45

To examine the products of combustion of a candle and of Coal-gas.—Arrange an apparatus like that in the



diagram, where A is a candle burning under an inverted filter funnel, connected first with an empty bulb-tube B, next with a boiling-tube C containing Lime-water, and lastly with an aspirator D to draw air through the whole apparatus. Notice that after a time a liquid collects in B,

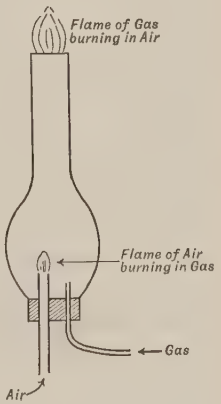
which, on being tested, turns Anhydrous Copper Sulphate blue, and must therefore be water; and at the same time the Lime-water in C is turned milky, proving that Carbonic acid gas is given off. Hence when a candle burns, the products of combustion are Water and Carbonic acid gas.

Now the substance which produces water when it burns is Hydrogen, and that which produces Carbonic acid gas is Carbon: therefore both these substances are present in the wax of the candle.

Next substitute a small jet of Coal-gas for the candle, having first dried the bulb-tube by blowing hot air through it, and put some fresh Lime-water into C. Prove that exactly the same products of combustion are obtained from a Coal-gas as from a candle flame.

Experiment 46

To show that air can be made to burn.—When the combustible substance and the supporter of combustion are both gases, their places may be interchanged: for example, Coal-gas ordinarily burns in air; hence air can be made to burn in Coal-gas. Arrange a wide-mouthed lamp-glass with a cork having two tubes through it, as in the figure. Turn on the gas, which burns in air at the top. Now push a lighted taper carefully through the tube A: a flame is seen inside the lamp-glass, which must be air burning, for it goes out if the supply of air be cut off by placing the finger over the open end of the tube.



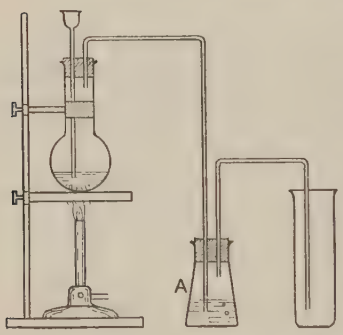
CHAPTER VI

THE HALOGENS AND THEIR ACIDS

Experiment 47

To prepare Chlorine and examine some of its properties.—N.B.—Do this in the draught cupboard, and take the very greatest care to avoid inhaling the gas.

Fit up a flask with safety funnel and delivery tube, as in the diagram. Arrange the apparatus so that the gas is bubbled through water in the bottle A, and if required dry, it may also be passed through strong Sulphuric acid contained in a second similar bottle. In the flask place enough Manganese Dioxide to form a layer about half a centimetre deep. Pour in enough strong Hydrochloric acid to cover the layer, and warm gently. The gas will be seen coming off, as it is greenish yellow in colour, and, being about two and a half times as heavy as air, is collected by downward displacement. Collect five jars of the gas, and then place the delivery tube into a large boiling-tube containing slaked Lime.



Perform the following experiments with the gas.

(1) Plunge a lighted taper into the gas: observe that the gas does not itself take fire, nor does it put the taper out; but the flame becomes faint, and a good deal of Carbon in the form of soot is deposited in the jar.

The wax of the taper consists largely of Carbon and Hydrogen, and the latter combines very readily with the Chlorine, leaving the Carbon to be deposited as smoke or soot.

(2) Moisten a filter-paper with Turpentine, wipe off any excess, and drop the paper into a jar of the gas. Turpentine consists of Carbon and Hydrogen; the latter combines with the Chlorine, setting free the Carbon, and so much heat is evolved that the paper often takes fire.

(3) Drop a pinch of Antimony powder into a jar of the gas.

(4) Drop into a jar of the gas a piece of Dutch metal (copper used for imitation gilding) and a wet postage-stamp.

(5) Place a small piece of Phosphorus in a deflagrating spoon, and plunge it into the gas.

Note carefully what you observe in each case in your notebook.

(6) Now collect one more jar of the gas, but in this case take care that it is as dry as possible. You can do this by bubbling it through Sulphuric acid, or by shaking up some powdered Calcium Chloride in a jar of the gas for a few minutes.

Drop into the gas a piece of dry red twill, and leave it for a short time. If the gas is quite dry, it will produce no effect. Now sprinkle a few drops of water on to the twill, and you will observe that the colour is bleached to a yellowish tint wherever the water has fallen. Also drop in

a piece of moist printed paper, and notice that the ink is not affected. Compare the result in the case of the postage-stamp. Try also whether ordinary writing-ink is affected. Now take the bottle containing water used to wash the gas. You will find that the water in it is saturated with Chlorine and has assumed the greenish colour of the gas, and also its irritating smell.

N.B.—Be very cautious how you smell the solution. You had better pour a little into a test-tube and dilute it with water first.

$$MnO_2 + 4HCl = MnCl_2 + 2H_2O + Cl_2.$$

We may also prepare Chlorine by the action of Sulphuric acid on Sodium Chloride mixed with Manganese Dioxide. Use the same form of apparatus. About 40 grms. of Sodium Chloride (common salt) mixed with 30 grms. of the black Oxide of Manganese are placed in the flask. Pour strong Sulphuric acid, diluted with about two-thirds of its volume of water, down the funnel, and warm gently. Chlorine will be given off.

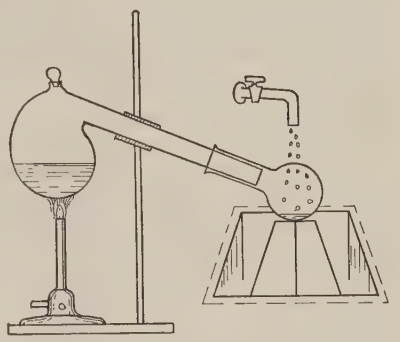
$$2NaCl + 2H_2SO_4 + MnO_2 = Cl_2 + Na_2SO_4 + MnSO_4 + 2H_2O.$$

N.B.—In diluting strong Sulphuric acid with water, pour the acid into the water slowly, a little at a time, stirring well, and keeping the mixture cool by placing the vessel containing it in water. If the water is poured into the acid serious results may follow.

Experiment 48

To prepare Bromine.—Place a mixture of Potassium Bromide and Manganese Dioxide in a small retort provided with a stopper; arrange a small flask over the end of

the retort, and keep it cool with a stream of water, as in the diagram. Now pour a little strong Sulphuric acid into the retort, replace the stopper, and heat gently. Bromine is given off as a red gas, and condenses to a reddish black liquid in the flask. When you have collected a small quantity, stop heating the retort and remove the flask. Pour two or three drops of the Bromine into a test-tube and shake it up with water. It dissolves, but not so easily as Chlorine. The solution will bleach in the same way as Chlorine, but less vigorously.



Warm a little Potassium Bromide, Manganese Dioxide, and Sulphuric acid in a test-tube. Bromine gas is given off; avoid inhaling it. Test it with a lighted splinter. The light goes out, and the gas does not burn. Hold a piece of moist litmus paper in the gas and notice the bleaching effect.

$$2KBr + 2H_2SO_4 + MnO_2 = Br_2 + MnSO_4 + K_2SO_4 + 2H_2O.$$

Experiment 49

To prepare Iodine.—Use the apparatus as in the preparation of Bromine, but make your mixture with Potassium Iodide instead of Bromide. On heating, violet vapours are given off, which condense to a dark grey shining solid. This is Iodine.

Shake up a little Iodine with water in a test-tube. The water becomes slightly yellow. If you add a little Potas-

sium Iodide solution to the water, the Iodine will dissolve more easily.

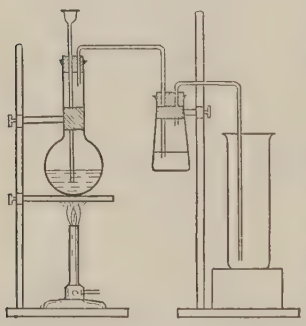
Add a little Starch paste to a dilute solution of Iodine. Note the violet colour produced; this forms an exceedingly delicate test for Iodine.

$$2KI + 2H_2SO_4 + MnO_2 = I_2 + MnSO_4 + K_2SO_4 + 2H_2O.$$

These three substances, Chlorine, Bromine, and Iodine, together with a fourth, Fluorine, form a group of elements known as the "Halogens." The preparation of Fluorine is attended with great difficulties, and requires special apparatus.

Experiment 50

To prepare Hydrogen Chloride or Hydrochloric acid gas.—N.B.—Do this in the draught cupboard.



Set up the apparatus as in the diagram. In the flask place a layer of common salt about 2 cms. deep. See that the lower end of the funnel nearly reaches the bottom of the flask, and that it is not choked with the salt. Pour strong Sulphuric acid through the funnel till it just covers the lower end. Warm gently, when the gas will soon be given off. It may be bubbled through strong Sulphuric acid to dry it, and collected by downward displacement, as it is a little heavier than air. Collect three jars full, and make the following observations: —

(1) It is colourless, but fumes or "steams" when allowed to escape into the air.

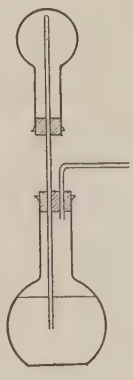
(2) It is acid (turns blue litmus red), will not support the combustion of a burning taper, nor burn itself.

(3) A glass rod dipped in strong Ammonia and brought near the mouth of the jar produces white fumes.

(4) Pour a little Silver Nitrate solution into a jar of the gas, and stand it in a strong light near a window for some time. Note what happens.

(5) Take a small flask and fit it with a cork having a straight tube passing through it, projecting up for about 6 or 8 in. Pour a little strong Ammonia into this, replace the cork, and heat the flask. Ammonia gas is given off, and collected by upward displacement in a gas-jar held inverted over the tube. When the jar is full, close the mouth with a plate, and over it, mouth to mouth, place a jar of Hydrochloric acid gas. Now slide out both cover-glasses. Dense white fumes of Ammonium Chloride will be formed, and small white solid particles of it may be seen clinging to the sides of the jars.

Hydrogen Chloride is very soluble in water. We may make a solution by attaching a small funnel to the end of the delivery tube and allowing its lower edge to dip into water in a beaker.



We may also show the solubility of the gas in the following striking way:—

Fit up two flasks as in the diagram. Half fill the lower flask with water coloured with blue litmus solution. Remove the upper flask from the cork and fill it with Hydrochloric acid gas, and replace it on the cork on the tube. Now blow into the lower flask through the short tube, so as to force the water just up to the top of the jet. The gas

will dissolve so rapidly that the water will spurt up in a little fountain, turning red at the same time.

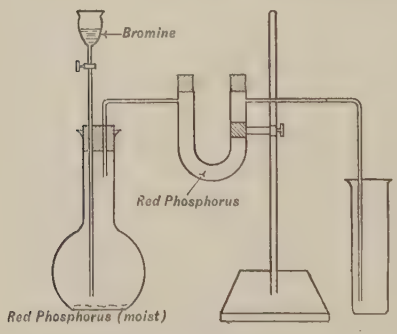
$$\left.\begin{aligned} NaCl + H_2SO_4 &= HCl + NaHSO_4 \\ NaCl + NaHSO_4 &= HCl + Na_2SO_4 \end{aligned}\right\}$$

Experiment 51

To prepare Hydrogen Bromide, or Hydrobromic acid gas.—If we try to prepare Hydrogen Bromide in the same way as we prepared the Chloride, only using Potassium Bromide instead of Sodium Chloride, we shall find that we get free Bromine as well as Hydrogen Bromide. The reason for this is that the Sulphuric acid acts on the Hydrogen Bromide as soon as it is formed in the flask, and decomposes part of it, liberating Bromine.

To obtain Hydrogen Bromide free from Bromine vapour, proceed as follows:—

Fit a flask with a dropping-funnel and delivery tube as in the diagram, and connect it to a U-tube containing red Phosphorus, moistened with a little water. In the flask put a thin layer of red Phosphorus, and just cover it with water. In the funnel put a little Bromine. Allow the Bromine to enter the flask very slowly, a drop at a time. Hydrogen Bromide is given off, and is freed from any trace of Bromine vapour



in passing through the U-tube. Collect in jars by downward displacement.

The gas strongly resembles Hydrogen Chloride, and the same experiments may be repeated with it.

$$2P + 3Br_2 = 2PBr_3 : 2P + 5Br_2 = 2PBr_5.$$
$$PBr_3 + 3H_2O = H_3PO_3 + 3HBr : PBr_5 + 4H_2O = H_3PO_4 + 5HBr.$$

Experiment 52

To prepare Hydrogen Iodide, or Hydriodic acid gas.—The method of preparation is the same as in the case of the Bromide, except that the Phosphorus and Iodine are mixed dry in the flask, and water is dropped from the funnel. The same characteristics, both physical and chemical, will be observed.

$$2P + 5I_2 = 2PI_5.$$
$$PI_5 + 4H_2O = H_3PO_4 + 5HI.$$

Experiment 53

To prepare Hydrogen Fluoride, or Hydrofluoric acid gas.—Do not attempt to collect this gas; but you can illustrate one of its remarkable properties as follows:—

Take a small shallow dish of Lead or Platinum, and put in it a little powdered Fluor Spar (Calcium Fluoride). Add as much Sulphuric acid (strong) as will make the powder into a thin paste. Melt some beeswax in an evaporating dish, and coat one side of a watch-glass with the warm wax. When cold, scratch your initials through the wax with a pin or the point of a penknife. Very gently warm the dish containing the paste, and expose the waxed side of the watch-glass to the fumes which rise from it, holding the

glass in a pair of tongs. After some time, say about five minutes, remove the watch-glass, and scrape or melt off the wax. You will find your initials etched into the glass.

N.B.—Be very careful to avoid inhaling the fumes of the gas—it is most poisonous; and do not allow the acid in any form to come in contact with your fingers.

$$CaF_2 + H_2SO_4 = CaSO_4 + 2HF.$$

CHAPTER VII

NITROGEN AND SOME OF ITS IMPORTANT COMPOUNDS

Experiment 54

To prepare Nitrogen.—We have already obtained Nitrogen, by depriving the air of its Oxygen in various ways. The best method for collecting the gas is to pass air very slowly over red-hot copper, contained in a hard glass or iron tube, which is heated in a furnace. Collect the gas over water in the pneumatic trough. The air must be passed into the tube containing the hot copper very slowly, and this is best done by allowing a small stream of water to enter the vessel containing the air.

Nitrogen is a colourless gas, without smell, and is slightly lighter than air. It will not burn or support combustion; it is, in fact, singularly inactive. All the usual tests give negative results. It can, however, be made to combine with Oxygen by passing sparks from a coil through a mixture of the two gases, and it also forms compounds called Nitrides with certain metals, e.g. Magnesium.

You may also prepare Nitrogen by heating a mixture of Sal-Ammoniac and Sodium Nitrite. Heat in a flask fitted with funnel and delivery-tube, and collect the gas over water in the usual way.

The reaction is in two stages, as shown by the equations given below. Ammonium Nitrite is first formed, and this, on being heated, decomposes into Nitrogen and water.

$$NaNO_2 + (NH_4)Cl = (NH_4)NO_2 + NaCl.$$
$$(NH_4)NO_2 = N_2 + 2H_2O.$$

Experiment 55

To prepare Nitric acid.—Place about 15 grms. of Potassium Nitrate (Saltpetre or Nitre) in a stoppered retort. Slip a small flask over the neck of the retort, and keep it cool by allowing cold water to run over it.

Pour strong Sulphuric acid into the retort, so as just to cover the Nitre: replace the stopper and heat gently. Brownish yellow fumes will fill the retort, and will condense in the flask to a yellow liquid.

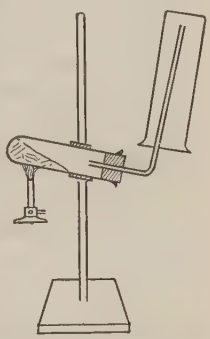
Do not continue the heating too long, or you will simply distil over Sulphuric acid: stop when about 10 c.c. of liquid are condensed. Examine this liquid; test it with litmus: try its effect on copper turnings in a test-tube; add a few drops of it to a solution of indigo.

Show also that Nitric acid will act as a supporter of combustion, by boiling a little strong Nitric acid in a large flask, and plunging Phosphorus, burning in a deflagrating spoon, into the vapour.

$$2KNO_3 + H_2SO_4 = K_2SO_4 + 2HNO_3.$$

Experiment 56

To prepare Ammonia and examine some of its properties.—Half fill a boiling-tube, fitted with a cork and bent delivery-tube as in the diagram, with a dry mixture of Sal-Ammoniac and about one and a half times its bulk of powdered Lime. Support the tube in a clamp with its mouth turned slightly downwards. Some water is formed in the action which takes place, and if allowed to run into the hot part of the tube will certainly crack it. Heat gently, moving the flame along the tube, and beginning nearest the cork. Collect the gas by upward displacement. Fill two jars and a small flask.



Perform the following experiments:—

(1) Test the gas with red litmus.

(2) Try the effect of a lighted taper plunged into a jar of the gas.

(3) Try to pour the gas upwards, as you did with Hydrogen.

(4) Show the solubility in water and also the action on red litmus, by repeating the "fountain" experiment, which you did with Hydrochloric acid gas.

$$CaO + 2(NH_4)Cl = CaCl_2 + 2NH_3 + H_2O.$$

Experiment 57

To prepare Nitrous Oxide and examine some of its properties.—Fit up a small retort with a delivery-tube (which should be rather wide) leading to a pneumatic trough containing hot water. N.B.—Heat the water before pouring it into the trough. In the retort put about 20 grms. of Ammonium Nitrate, and heat cautiously with a small flame. Collect five jars of the gas, and perform the following experiments:—

(1) Plunge a glowing splinter into the gas.

(2) Try the effect of plunging (*a*) burning Sulphur, (*b*) burning Phosphorus (yellow and also red) into the gas. Try first with the Sulphur and Phosphorus only burning feebly, and afterwards try with both well alight. In each case test the gas left after the combustion with a lighted taper.

(3) Test the solubility of the gas in cold water, by shaking up a jar of the gas after pouring in some water, and notice if the cover-glass sticks to the mouth.

(4) Note whether the gas has any smell.

Nitrous Oxide is called "Laughing Gas"—if breathed in large quantities it produces unconsciousness.

$$(NH_4)NO_3 = N_2O + 2H_2O.$$

Experiment 58

To prepare Nitric Oxide, and examine some of its properties.—Cover the bottom of a flask with a layer of Copper turnings, and pour about 50 c.c. of Nitric acid diluted with an equal volume of water down the funnel. If the gas is not evolved in a few moments, warm gently. Brown fumes appear in the flask: these are not Nitric Oxide, but another Oxide of Nitrogen formed by the combination of the Nitric Oxide as it comes off with the Oxygen of the air in the flask. After a time the air is exhausted, the brown colour disappears, and the gas may then be collected over water. Collect three jars of the colourless gas, and examine it as follows:—

(1) Uncover one jar to admit the air; notice the brown fumes.

(2) Introduce a lighted splinter into a second jar.

(3) Plunge a deflagrating spoon containing yellow Phosphorus—(*a*) burning feebly, (*b*) burning briskly—into the third jar. When the Phosphorus has burnt, and the white fumes disappeared, test the gas left with a burning taper.

(4) Filter off the green solution left in the flask, concentrate it by boiling away about half in an evaporating dish, and set it aside for crystals to form. These are Copper Nitrate.

$$3Cu + 8HNO_3 = 2NO + 3Cu(NO_3)_2 + 4H_2O.$$

CHAPTER VIII

SULPHUR—ITS VARIOUS FORMS, AND SOME OF ITS IMPORTANT COMPOUNDS

Sulphur is one of the elements which exist in what are known as "Allotropic" forms; that is to say, it exists in varieties having physical characteristics which differ widely from one another, but whose chemical properties are identical. Phosphorus is another such element. We have already seen two forms of this.

Experiment 59

To prepare and examine the different forms of Sulphur.—Crush about 5 grms. of roll Sulphur in a mortar to a fine powder. Place it in a test-tube, add about half a tube full of Carbon Disulphide, and shake vigorously. Filter, and collect the filtrate in a beaker, cover it with a filter-paper, and stand it in the draught cupboard. Crystals will form. Separate them from any liquid, dry them on filter-paper, and examine them with a lens, noting the shape. Make a sketch in your notebook. These are "Rhombic" Sulphur, sometimes called α Sulphur. Keep some of the crystals.

N.B.—Carbon Disulphide is *highly* inflammable. No flame must be allowed to burn anywhere near it.

Melt a fair-sized lump of roll Sulphur in a porcelain dish till it is quite liquid, but avoid overheating it. Allow it to

cool, and as soon as a crust is formed on the top, break it through by making a small hole at both sides of the dish, and pour out the liquid below it into a dish of cold water, holding the dish in the tongs. When cold, break away the crust, and examine the Sulphur below it. Notice the shape of the crystals, and make a sketch in your notebook. This is "Monoclinic" or "Prismatic" Sulphur, sometimes called β Sulphur. Keep some of the crystals.

Heat Sulphur to near its boiling-point in a test-tube, and pour it suddenly into cold water. Take it out, wipe it, and examine it. This is "plastic" Sulphur, sometimes called γ Sulphur. Put one piece aside for a day or two, and then examine it again. Keep another piece.

Take the specimens of rhombic, monoclinic, and plastic Sulphur that you have kept, and try which of them is soluble in Carbon Disulphide.

You may further try to find (*a*) the density, (*b*) the melting-point of α and β Sulphur.

Tabulate your results.

Experiment 60

To prepare Sulphuretted Hydrogen.—Place a few lumps of Ferrous Sulphide in a flask fitted with funnel and delivery tube. Pour dilute Hydrochloric acid down the funnel, bubble the gas through water, and collect by downward displacement. Collect several jars, and observe:—

(1) The effect on a lighted taper.

(2) The effect of shaking up the gas with water. Test the solution with litmus.

(3) Try the effect of burning the gas at a fine jet about 20 cms. long, attached to the delivery-tube. Hold a cold glass tube filled with water in the flame. Heat the tube of

the jet about 15 cms. from the burning gas. Record your observations.

(4) Moisten a piece of filter-paper with a solution of Lead Nitrate, extinguish the burning gas, and allow it to escape on to the moist paper. The dark stain produced is Lead Sulphide. This forms a delicate test for the presence of Sulphuretted Hydrogen.

(5) Mix jars full of Sulphuretted Hydrogen and Sulphur Dioxide; note the deposit of Sulphur.

(6) Bubble the gas through solutions of Copper Sulphate, Cadmium Sulphate, Antimony Chloride, and Iron Chloride, contained in small test-tubes. Note the coloured deposits formed. These are called "Precipitates"; they consist of the insoluble Sulphides of the metals.

Sulphuretted Hydrogen is often prepared in a "Kipp's Apparatus." If there is one in the laboratory, examine it and try to understand the principle on which it is arranged.

$$FeS + 2HCl = H_2S + FeCl_2.$$

Experiment 61

To prepare Sulphur Dioxide and examine some of its properties.—This gas is formed when Sulphur burns in air or Oxygen. It is usually prepared in the laboratory as follows:—

Place a layer of copper turnings in a flask fitted with thistle funnel and delivery-tube; pour strong Sulphuric acid down the funnel, and warm the flask gently. Remove the flame or turn it low when the action begins. Collect the gas by downward displacement in several jars, and examine it as follows:—

(1) Test it with a lighted taper.

(2) Shake a jar of it up with a little water, and test the solution with blue litmus.

(3) Fill a flask with it, and do the "fountain" experiment.

(4) Bubble some of the gas through a solution containing a little magenta dye: note its bleaching effect.

(5) Test a jar of the gas by holding in it a piece of filter-paper which has been dipped in a solution of Potassium Bichromate; note that it is turned green. This forms a very delicate test for Sulphur Dioxide.

(6) Pass the gas for three or four minutes through a solution of Potassium Hydrate: note that it gets hot, showing that chemical action is taking place. Evaporate the solution and prepare crystals: these are Potassium Sulphite.

(7) Filter off some of the liquid left in the flask, and try to prepare crystals from it. These are Copper Sulphate.

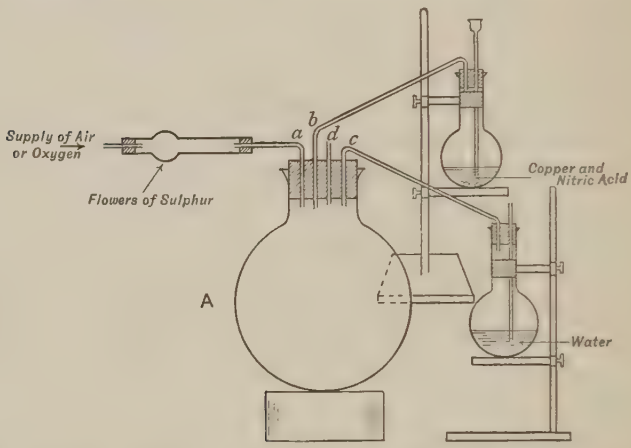
$$Cu + 2H_2SO_4 = SO_2 + CuSO_4 + 2H_2O.$$

Experiment 62

To prepare Sulphuric acid.—Fit up the apparatus shown in the diagram. A is a large flask (4 or 5 litres), provided with a rubber cork having four tubes—*a*, *b*, *c*, *d*—leading into it. The tube *a* is connected with a bulb-tube containing Flowers of Sulphur, connected at the farther end with a foot-bellows, so that air can be sent through it; or better, joined up to a supply of Oxygen. The tube *b* is connected with a flask for generating Nitric Oxide (see Experiment 58) from copper turnings and Nitric acid; *c* is connected to a flask containing water, so that steam can be passed into A.

First let some steam enter A. Then heat the Sulphur

till it burns in the air or Oxygen passing over it, and at the same time pass in some Nitric Oxide. After some time you



will observe drops of liquid condensing on the sides of A. This is Sulphuric acid. You may consider the following to be what takes place.

Nitric Oxide meets with Oxygen from the air in A, and is oxidized to Nitrogen Peroxide (brown fumes).

$$NO + O = NO_2.$$

This Nitrogen Peroxide gives up half its Oxygen to the Sulphur Dioxide which enters through the tube *a*, and is itself reduced again to Nitric Oxide.

$$SO_2 + NO_2 = SO_3 + NO.$$

This Nitric Oxide is ready to take up more Oxygen, and repeat the above process.

The Sulphur Trioxide SO_3 combines with the steam to form Sulphuric acid.

$$SO_3 + H_2O = H_2SO_4.$$

When a small quantity of liquid has collected at the bottom of A, disconnect the apparatus, pour out the liquid into a beaker, and examine it as follows :—

(1) Test it with litmus.

(2) Pour a few drops of it into a small quantity of water in a test-tube. Write your name on paper with this solution; the letters will be invisible till you warm the paper, when they will appear black.

(3) Add a few drops of dilute Sulphuric acid to a small quantity of Barium Chloride solution in a test-tube. You will get a white precipitate of insoluble Barium Sulphate. Now try whether you get the same result with the acid you have prepared.

Experiment 63

Try the effect of—

(1) Dilute Sulphuric acid on zinc.

(2) Strong Sulphuric acid on zinc, cold and hot.

Try both these in a test-tube, and identify the gases given off.

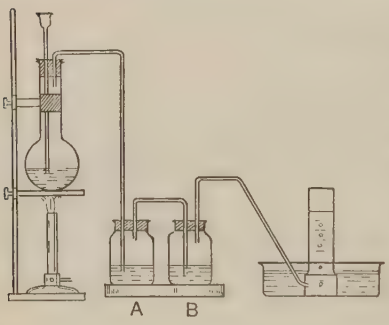
Try also the effect of Sulphuric acid on Copper Sulphate crystals.

CHAPTER IX

COMPOUNDS OF CARBON

Experiment 64

To prepare Carbon Monoxide and examine some of its properties.—Set up the apparatus as in the diagram, and see that the connections are gas-tight. In the flask put about 15 grms. of Oxalic acid crystals, and in the two bottles A and B put a strong solution of Caustic Soda. Now pour down the funnel enough strong Sulphuric acid to cover the crystals, and heat gently. Try to make the gas come off as slowly as possible, and take the greatest care not to breathe it, as it is very poisonous. The gas given off is a mixture of Carbon Monoxide and Dioxide, but the latter will be absorbed by the Caustic Soda solution, if the gas does not bubble through too fast. Collect some jars of the gas, and examine it as follows:—



(1) Shake it up with Lime-water.

(2) Place a lighted taper to the gas.

(3) Try if the gas is soluble in water.

(4) Try to find out what is formed when the gas burns in air.

$$(COOH)_2 + H_2SO_4 = CO + CO_2 + H_2SO_4 . H_2O.$$

Experiment 65

To prepare Methane, or Marsh gas.—Make a mixture of Anhydrous Sodium Acetate and Soda-Lime in the proportion of 1 to 2, by weight. Put the mixture into a hard glass tube, fitted with cork and delivery-tube, and heat, collecting the gas which comes off over water. The gas is Methane. Fill several large test-tubes with it, and examine it as follows :—

(1) Apply a lighted taper to the mouth of one of the tubes.

(2) Test the gas with Lime-water. Now burn it, and test again.

(3) Try to pour the gas upwards.

$$CH_3 . COONa + NaOH = Na_2CO_3 + CH_4.$$

Experiment 66

To prepare Ethylene.—Take about 15 c.c of Methylated spirit in a large beaker, and slowly add to it about 80 c.c. of strong Sulphuric acid, keeping the beaker cooled with water while you do it.

Pour the mixture into a flask fitted with funnel and delivery-tube, and heat it gently. Ethylene gas will be given off, and may be collected over water. Collect two or three test-tubes full, and apply the tests as for Methane.

$$\begin{cases} C_2H_5OH + H_2SO_4 = C_2H_5HSO_4 + H_2O. \\ C_2H_5 . HSO_4 \quad\quad = C_2H_4 + H_2SO_4. \end{cases}$$

Experiment 67

To prepare Acetylene.—N.B.—Before doing this experiment, ask your master which method to use.

(1) Place some pieces of Calcium Carbide in a flask, fitted with a dropping funnel and delivery-tube. Connect the end of the delivery-tube to the gas supply, and sweep all the air out of the flask with a current of gas. Now put some water into the funnel, and allow it to fall on the Carbide a few drops at a time. Collect the gas over water, but reject the first two or three jars. Notice that the gas burns quietly unless mixed with air, and observe also its peculiar smell. Now connect the delivery-tube by means of a piece of rubber tubing to a batswing burner, and light the gas. Hold a piece of porcelain in the flame. Allow the flame to play upon a large test-tube full of cold water. Observe what is left in the flask when the action has ceased.

(2) Place a small lump of Carbide in a porcelain crucible, and invert over it a thistle funnel with the stem cut off short. Fill a pneumatic trough with water and stand in it a gas-jar, inverted and also full of water. Now plunge the crucible and funnel under the water, and at once place the jar over them. The gas collects in the jar, and may be then safely burnt.

$$CaC_2 + 2H_2O = Ca(OH)_2 + C_2H_2.$$

CHAPTER X

THE LAWS OF CHEMICAL COMBINATION

We may state those laws as follows:—

(1) The Law of Definite Proportions.

The same compound, however it is made, always contains the same elements united in the same proportions by weight.

(2) The Law of Multiple Proportions.

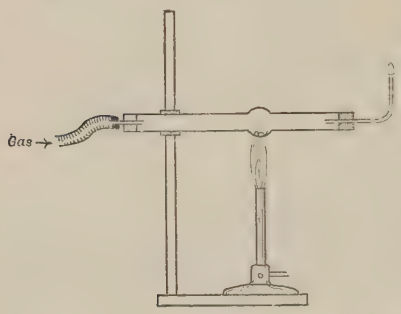
If one element forms more than one compound with another, the weights of one which combine with a constant weight of the other are in the proportion of simple whole numbers, or are simple multiples of a common factor.

We shall now perform experiments with a view to illustrate the truth of these laws. If good results are to be obtained, the materials used should be pure, and great care must be taken with the weighings and manipulation.

Experiment 68

To prepare the black Oxide of Copper in three different ways, and to show that its composition is definite and the same in each case.—Half fill a small porcelain crucible with some Copper Carbonate. On heating steadily on a tobacco-pipe triangle, the Carbonate will give off Carbon Dioxide and be converted into Cupric Oxide. In the same

way prepare Cupric Oxide by heating Copper Hydrate and Copper Nitrate. While these are getting cold, carefully weigh a hard glass bulb-tube, as in the diagram. Fold a narrow strip of paper, and slide it into the tube, making a channel down to the bulb. Slide about one gram of the black Oxide down the paper into the bulb. Weigh again. Fit up the tube as shown, and pass a gentle stream of Coal-gas over the Oxide, lighting the gas as it escapes from the tube. Now heat the Oxide, gently at first, and then strongly. It will be reduced to metallic Copper in the form of a reddish powder. When it has all turned colour, allow it to cool, still keeping the Coal-gas passing over it. Weigh again when cold.



Enter your results thus :—

	Grms.
Weight of tube, empty	
„ „ and Oxide	
„ Oxide taken	
„ tube and metallic Copper . .	
„ metallic Copper obtained . .	

From these observations find the weight of Copper which combines with 1 grm. of Oxygen, in each of the three specimens of Oxide prepared. It should be nearly the same in each case.

Hydrogen gives more accurate results than Coal-gas, but is more trouble, unless you have a supply in a gas-holder.

The Law of Multiple Proportions should now be illustrated by the following experiments.

Experiment 69

To reduce the red Oxide of Copper in a stream of Hydrogen or Coal-gas, and hence to find the weight of copper which combines with 1 grm. of Oxygen, and to compare it with the weight found in the case of the black Oxide.—The Oxide used must be pure. Perform the reduction exactly as in the last experiment. If your work is accurately done, you will find that the red Oxide contains twice as much Copper in proportion to the Oxygen as the black.

Experiment 70

To find the weight of Lead combined with 1 grm. of Oxygen in each of the three Oxides of Lead.—Cut out an oblong piece of thin sheet copper and fold it up so as to make a little boat which will slip easily into a tube of 1 cm. internal bore. Weigh it carefully; introduce about 2 grms. of Lead Monoxide, and weigh again. Slip the boat into a hard glass tube, pass Hydrogen over it, and heat. The Oxide will be reduced, leaving metallic Lead. Allow it to cool in the stream of gas, and when cold weigh it again.

From your observations calculate the weight of Lead which combines with 1 grm. of Oxygen.

Repeat the experiment with red Lead Oxide and with Lead Peroxide. Compare the weights of Lead. They should be approximately as 6 : 4 : 3.

If a small Carbon crucible and a muffle-furnace are available, the Oxides may be reduced in the crucible, and the weights of the Lead beads left found.

Experiment 71

To find the weight of Iron combined with 1 grm. of Sulphur in each of the two Sulphides of Iron.—Take about 1 grm. of each of the two Sulphides of Iron—Ferrous Sulphide and Iron Pyrites—well powdered, and reduce them separately, as in Experiment 68, by passing a stream of *dry* Hydrogen gas over them. Test the gas which is given off and try to identify it.

Calculate the weight of Iron which combines with 1 grm. of Sulphur in each case.

CHAPTER XI

MEASUREMENT OF VOLUMES OF GASES CORRECTIONS FOR TEMPERATURE AND PRESSURE

Since gases expand when heated, it is obvious that a given vessel will contain a smaller weight of a gas which is warm than it will of the same gas when cooler. Also since gases can be compressed, and expand again when the pressure is reduced, it follows that a given vessel contains a greater weight of a compressed gas than it does of the same gas under less pressure.

In order to compare volumes of gases collected under varying conditions of temperature and pressure, we must measure them under "Standard" or "Normal" conditions.

Normal Temperature is 0° C., and Normal Pressure is equivalent to that pressure which would support a column of Mercury 760 mm. long. N.T.P. or S.T.P.

These conditions will never be found in the laboratory, so we must measure the volume of the gas under existing conditions, observing what these are, and then apply "corrections" to obtain the "standard" or "normal" value.

RULES FOR CORRECTING VOLUMES OF GAS FOR TEMPERATURE AND PRESSURE

Observe the temperature at which the volume is actually measured. If the gas is collected over a liquid, this will be the temperature of the liquid; otherwise observe the temperature of the room. Suppose this is t° C. and V is the measured volume.

Then the standard volume is $V \times \frac{273}{273+t}$ if t is above 0° C., and $V \times \frac{273+t}{273}$ if t is below 0° C., which will seldom be the case.

The correction for temperature being now made, we proceed to correct for pressure as follows:—

Read the barometer in mm. of Mercury. Multiply the observed volume by this, and divide by 760.

Thus, given 150 c.c. of a gas collected at 12° C. and 752 mm., the standard volume is $150 \times \frac{273}{273+12} \times \frac{752}{760}$ c.c.

There is, however, another point to be observed. When a gas is collected over water, it is mixed with a certain amount of water-vapour, which of course exerts some pressure on the surface of the liquid. This pressure varies with the temperature, and tables are published showing the pressure in mm. of Mercury exerted by water-vapour at different temperatures (see p. 134).

This pressure must be subtracted from the pressure shown on the barometer before the correction is applied as above.

Experiment 72

To find the weight of a litre of Hydrogen.—Take a large, dry flask with a round bottom, and fit it with a rubber cork, having a short tube passing through it, closed with a clip. Remove the cork, and fill the flask with Hydrogen by upward displacement. Generate the gas in the usual way with zinc and acid, and dry it by passing it through a Calcium Chloride tube. When the flask is full, close it with the cork and clip, and weigh it very carefully. Now attach the flask to a pump, and exhaust as far as possible. Weigh again. Hold the flask so that the neck dips under water and open the clip. The water will rush in, nearly filling the flask. Adjust the levels so as to get the Hydrogen left inside at atmospheric pressure, close the clip, and remove the flask from the water. Measure the water in the flask with a graduated cylinder. Enter your results thus:—

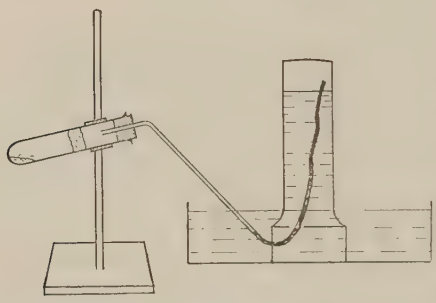
	Grms.
Weight of flask full of Hydrogen . . .	
„ „ partly exhausted . . .	
Volume of water entering flask . . .	
„ Hydrogen removed by pump .	
Weight of	

Hence find the weight of 1000 c.c.

A good result would be ·09 grm.

Experiment 73

To find the weight of a litre of Oxygen.—Take a hard glass test-tube, and fit a cork and delivery-tube, as in the diagram. Crush some Potassium Chlorate in a mortar, and put it into the bottom of the tube, and above it place a small wad of asbestos wool. Weigh the tube and contents without the cork. Attach a rubber tube B to the end of the delivery-tube, and insert it through a beehive shelf into a graduated gas-jar, as in the diagram. Now heat the Chlorate, so as to drive off Oxygen. Do this cautiously, so as not to fill the cylinder entirely. When it is nearly full stop the action, and cool the cylinder A by pouring water over it. When cold, slip out the tube B, put a plate over the mouth of the cylinder, and transfer it to a deep vessel of water to adjust the level inside and out. Read the volume of the gas, and make the usual corrections for temperature, pressure, and water-vapour. Remove the cork from the test-tube, and when cold weigh the tube and contents again. The loss of weight represents the weight of the Oxygen collected, and from this is found the weight of 1000 c.c.



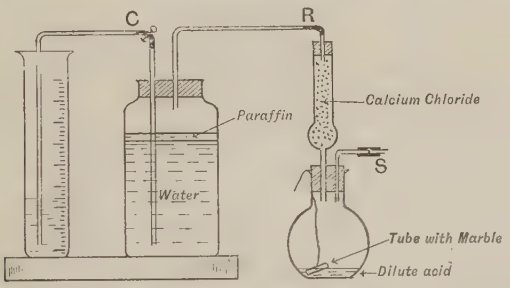
N.B.—You have collected in the cylinder a small quantity of air as well as Oxygen. This air was contained in the tubes before the heat was applied. But these tubes are now full of Oxygen, so that the error is the difference between the weights of air and Oxygen which the tubes would contain. This is small enough to be neglected.

A good result would be near 1·44 grms.

Experiment 74

To find the weight of a litre of Carbon Dioxide.—Fit up apparatus as in the diagram, taking care to see that all joints are gas-tight.

The small tube contains about a gram of powdered marble, and the flask contains dilute Hydrochloric acid. Before starting the action by allowing the acid to come in contact with the marble, weigh the whole apparatus below the rubber joint R. Now connect R, and tilt the flask till the action begins. Carbon Dioxide is given off, and is dried by the Calcium Chloride: it displaces the water in the aspirator, but is prevented from dissolving in it by the layer of paraffin. When all action has ceased, close the clip C, and



let the water in the tube to the left of C run into the graduated cylinder. Read the volume of water in the cylinder. Disconnect R, open the stopper S, and suck out the gas left in the flask and tubes.

Weigh as before. The loss of weight represents the weight of Carbon Dioxide given off, and the volume of this gas at the temperature of the water is given by the volume of water which has been driven out into the cylinder. Correct this volume for temperature and pressure, and hence calculate the weight of 1000 c.c. of Carbon Dioxide.

A good result would be near 1·98 grms.

CHAPTER XII

EQUIVALENT WEIGHTS

The Equivalent Weight (or Equivalent) of an element is that weight of it which combines with or displaces 1 grm. of Hydrogen.

Experiment 75

To find the equivalent of Magnesium by direct displacement of Hydrogen.—Magnesium displaces Hydrogen from Hydrochloric or Sulphuric acid. We shall measure the volume displaced by a known weight of Magnesium. Then, since we know the weight of a litre of Hydrogen, the weight of the displaced Hydrogen can be found, and hence the weight of Magnesium which displaces 1 grm. of Hydrogen.

Take a clean piece of Magnesium wire or ribbon and weigh it carefully. The quantity taken depends on the size of the graduated gas-cylinders which you have. For a cylinder taking 300 c.c. you must not have more than 0·26 grm. of Magnesium. Place the Magnesium in a small test-tube, and fill it with water.

Now fill the graduated cylinder with dilute acid, and invert it in a pneumatic trough, also containing acid. Raise the cylinder slightly, and slip the tube and Magnesium under it, and stand the cylinder down again.

When all action appears to have ceased, and the Magnesium has all disappeared, slip a plate over the mouth of the cylinder and remove it to a deep vessel of water. Take away the plate, and raise or lower the cylinder till the level of the liquid is the same inside and out. Read the volume of the gas: note also the temperature of the acid and the height of the barometer. Ascertain the pressure of water-vapour corresponding to the observed temperature, and enter your results thus:—

Weight of Magnesium used . . .	grms.
Barometer reading	mm.
Thermometer	C.
Pressure of water-vapour at C. . .	mm.
Volume of Hydrogen under these conditions	c.c.

Apply the corrections and calculate the volume the Hydrogen would occupy at S.T.P.

Also calculate its weight, assuming that a litre of Hydrogen weighs ·09 grm.

Hence calculate the weight of Magnesium which would displace 1 grm. of Hydrogen.

Experiment 76

To find the equivalent of Zinc.—Proceed exactly as in the case of Magnesium. Use Hydrochloric acid, and Zinc as clean and pure as possible. Do not use more than ·8 grm. for a 300 c.c. cylinder.

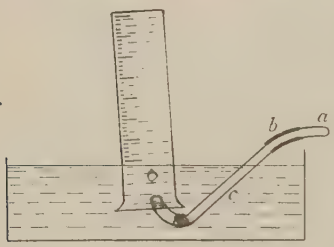
Experiment 77

To find the equivalent of Aluminium.—Proceed as in the last experiment, using Aluminium and dilute Hydrochloric acid. Take about ·2 grm. for a 300 c.c. cylinder; the action will probably be very slow, in which case add to the acid a few drops of Platinic Chloride.

Enter results and make calculations as before.

Experiment 78

To find the equivalent of Sodium.—We cause Sodium to displace Hydrogen from Methylated spirit as the action is less violent than in the case of water. The apparatus shown in the figure should be used. The tube *a* contains the weighed piece of Sodium, *b* is a joint of rubber gas-tubing, *c* is a bent glass tube of fairly wide bore passing under the graduated cylinder. By slightly raising *a*, the Sodium is caused to slide down through *b* and *c*, and at once rises up through the spirit in the cylinder.



The Sodium must be cut and weighed as rapidly as possible just before being used. It is best to weigh the tube *a* empty, with a cork, then put the Sodium in it, cork the tube, and weigh again. Take ·6 grm. for a 300 c.c. cylinder.

Enter results and make calculations as before, but take the pressure of the vapour of Methylated spirit to be four times that of water at the same temperature.

Experiment 79

To find the equivalent of Zinc, by causing it to displace Copper from a solution of Copper Sulphate.—Weigh a small porcelain dish, and then place in it about ·8 grm. of clean Zinc foil. Weigh again.

Half fill the dish with a solution of Copper Sulphate, and stir with a glass rod, warming slightly to accelerate the action. If the solution loses its colour, add more. When the Zinc seems to have all disappeared, allow the Copper to settle, and carefully suck off as much liquid as possible with a pipette. Wash the Copper three or four times, using a wash-bottle and sucking off the liquid as before. Wash the stirring-rod into the dish. Now wash twice with a little Alcohol, and drain off. Dry the Copper in the dish over a sand-bath, being careful not to overheat it: and when cold, weigh again.

Enter your results thus:—

	Grms.
Weight of porcelain dish, empty . . .	
„ „ „ and Zinc . .	
„ Zinc taken	
„ dish and Copper	
„ Copper precipitated . . .	

But 31·5 grms. of Copper are equivalent to 1 grm. of Hydrogen. Hence find the weight of Zinc which is equivalent to 1 grm. of Hydrogen. Compare your result with that previously obtained.

Experiment 80

To find the equivalent of Lead, by causing Zinc to displace Lead from a solution of Lead Acetate.—Proceed as in the last experiment, using a solution of Lead Acetate instead of Copper Sulphate.

Taking the equivalent of Zinc as 32·5, calculate that of Lead.

Experiment 81

To find the equivalent of Silver, assuming that of Chlorine to be 35·5.—N.B.—Do this in the draught cupboard.

Carefully weigh a glass tube with a bulb in the centre. Introduce by means of a folded slip of paper about ·5 grm. of finely divided Silver into the bulb, and weigh again.

Now pass a stream of dry Chlorine over the Silver. Prepare the Chlorine as in Experiment 45, and arrange the apparatus so that the escaping gas is absorbed by slaked Lime or dissolved in a beaker of water.

Enter your results thus:—

	Grms
Weight of bulb-tube, empty	
„ „ „ and Silver . . .	
„ Silver used	
„ tube and Silver Chloride . .	
„ Silver Chloride obtained . .	

From these observations calculate the weight of Silver which combines with 35·5 grms. of Chlorine, and consequently with 1 grm. of Hydrogen.

You can also find the equivalent of Silver by reducing a known weight of Silver Chloride in a stream of Coal-gas. Proceed exactly as when reducing Copper Oxide. (See Experiment 66.)

Experiment 82

To find the equivalent of Tin.—Carefully weigh a small porcelain crucible with its lid. Introduce about ·5 grm. of pure Tin-foil, and weigh again.

Take a piece of glass tube about 6 in. long, and draw out a jet at one end. Dip this jet into strong Nitric acid, and place your finger over the other end. Now put the lid on the crucible, raise it slightly at one side, insert your glass tube, and allow one or two drops of acid to fall on the Tin. Close the lid, wait a minute or so, and repeat the process. After a time the addition of more acid causes no further action. Heat the crucible carefully till the contents are dry; then heat it very strongly for some time, say ten minutes. When cold, weigh it again. The Tin is converted into Tin Oxide, and the increase in weight is the weight of the Oxygen.

Enter your results thus:—

	Grms.
Weight of crucible and lid, empty . .	
„ „ „ and Tin . .	
„ Tin used	
„ crucible, lid, and Tin Oxide . .	
„ Oxygen combined with Tin . .	

But 8 grms. of Oxygen are equivalent to 1 grm. of Hydrogen. Hence calculate the equivalent of Tin.

Experiment 83

To find the equivalent of Magnesium by converting it into Oxide: (*a*) in air; (*b*) by Nitric acid.

(*a*) Proceed as in the last experiment, but convert the Magnesium into Oxide by heating the crucible with the lid on, raising the latter slightly from time to time to admit air.

(*b*) Proceed exactly as in the case of Tin.

Experiment 84

To find the equivalent of Sulphur, by causing it to combine directly with Copper.—Weigh a small porcelain crucible, empty, and also containing about half a gram of clean Copper filings. Now cover the Copper with a thick layer of Flowers of Sulphur, put on the lid, and heat strongly till the excess of sulphur is completely volatilized. Allow the crucible to cool, take off the lid, and weigh again. The increase in weight gives the weight of Sulphur which has combined with the known weight of Copper. Taking the equivalent of Copper as 31·5, calculate the weight of Sulphur which will combine with this weight of Copper, and consequently with 1 grm. of Hydrogen.

CHAPTER XIII

ACIDS, BASES, AND SALTS

Experiment 85

To compare the properties of the Oxides of non-metals and of metals.—Take small fragments of Carbon, Sulphur, and Phosphorus separately in three clean deflagrating spoons, set each alight and burn it in a separate gas-jar containing air (or better, Oxygen), having half an inch of water at the bottom.

By this means the Oxides of these three non-metals are prepared, and if, when combustion has ceased, the jars be well shaken with the water, solutions of their Oxides are obtained.

Test these three solutions (*a*) by placing the tip of the finger in the liquid and carefully tasting it, (*b*) with red litmus paper, (*c*) with blue litmus paper: and make a table of the results in your notebook. Notice that all three solutions have an acid taste, do not affect red litmus, but turn blue litmus red, and must therefore be acids. Hence it may be inferred that aqueous solutions of non-metallic Oxides are acid in character.

Next prepare the Oxides of the metals Zinc, Calcium, and Sodium, by taking a little of each in a clean iron spoon and heating it strongly in air till it catches fire; notice that white powders are formed in each case. (If no metallic Calcium is available, this part of the experiment may be

omitted, and Quicklime—Calcium Oxide—used instead.) Now shake up a little of each Oxide in a separate test-tube with distilled water, and test the three liquids obtained by tasting, and with blue and red litmus paper. Tabulate your results as before.

No effect is seen with the Zinc Oxide: this is because it is almost insoluble in water. The solution of Calcium Oxide has a slightly bitter taste, and turns red litmus blue, while that of Sodium Oxide has a very bitter taste, and is strongly alkaline.

Hence we infer that when metallic Oxides dissolve in water their solutions are alkaline. These solutions are called "Bases" or "Hydroxides," though the term "Base" is also applied to those metallic Oxides which are insoluble in water.

Experiment 86

To investigate the action of Acids on Bases, and to make a Salt.—Take a dilute solution of Sodium Hydroxide (Caustic Soda) in one boiling-tube, and a dilute solution of Sulphuric acid in another. Place about 20 c.c. of the alkaline solution in a porcelain dish, and add the acid to it gradually, testing from time to time with blue litmus paper by putting a drop of the liquid on to the paper with a glass rod. After a time the litmus will be turned red, showing that excess of acid has been added. Now add carefully, drop by drop, the alkaline liquid, till a stage is reached when the solution will affect neither blue nor red litmus. The liquid is now said to be "neutral," and the process is called "neutralization." Next evaporate the liquid in the dish over a Bunsen flame, till half has been boiled away. Allow the remainder to cool and crystallize, when colourless

crystals will separate out. The substance formed is called a "Salt," and its name in this case is Sodium Sulphate.

The equation for the reaction is:—

$$2NaOH + H_2SO_4 = Na_2SO_4 + 2H_2O.$$

Base and Acid produce Salt and Water.

This experiment may be repeated with different bases and acids; and from the results we infer that when mixed in the proper proportions they neutralize one another, forming Salts and Water.

When a basic Oxide is insoluble in water, Salts may usually be prepared from it by dissolving it in dilute acids.

Experiment 87

To prepare crystals of the Salt Lead Nitrate.—Take about 50 c.c. of dilute Nitric acid in a beaker, warm it gently, and add to it gradually powdered Lead Oxide (Litharge) till, on stirring well and warming still more, no more of the solid dissolves, and a turbid liquid is obtained. Filter this liquid into a porcelain dish, evaporate down to half the original bulk, and allow it to crystallize, when crystals of Lead Nitrate separate out.

The equation for the reaction is:—

$$PbO + 2HNO_3 = Pb(NO_3)_2 + H_2O.$$

Base and Acid produce Salt and Water.

Another convenient method for obtaining a Salt is by dissolving the Carbonate of the metal in the required acid, the Carbonic acid gas which is evolved being got rid of by gently warming the liquid.

Experiment 88

To prepare crystals of Copper Sulphate.—Take some dilute Sulphuric acid in a beaker, warm it a little, and then add Copper Carbonate gradually till there is no more effervescence of Carbon Dioxide, and a turbid liquid is obtained.

Filter this liquid into a porcelain dish, evaporate half of the filtrate away over a Bunsen flame, and allow the remainder to crystallize, when blue crystals of Copper Sulphate are obtained.

The equation for this reaction is :—

$$CuCO_3 + H_2SO_4 = CuSO_4 + H_2O + CO_2.$$

Acids which are dibasic—that is, which contain two atoms of Hydrogen in the molecule which can be replaced by monovalent metals, e.g. Sodium—possess the power of forming two series of salts: in one series both atoms of Hydrogen are replaced by atoms of the metal,—these are called Normal salts—in the other series only one of the Hydrogen atoms is replaced by the metal—these are called Hydrogen salts, or sometimes Acid salts.

Thus, using the dibasic Sulphuric acid and Caustic Soda, we can prepare Normal Sodium Sulphate, Na_2SO_4 (see Experiment 85), and Sodium Hydrogen Sulphate, $NaHSO_4$.

Experiment 89

To prepare a pure specimen of Sodium Hydrogen Sulphate.—Place the dilute Sulphuric acid in a burette, and take about 25 c.c. of dilute Caustic Soda solution in a porcelain dish; now add the acid to the alkali very carefully, testing constantly with litmus paper, till the point is reached when the blue litmus is very slightly reddened by the acid. At this point the liquid would, if evaporated, yield the Normal salt. Now notice on the burette exactly how many c.c. of the Sulphuric acid have been added, and add to the liquid in the dish a precisely equal amount; then evaporate to dryness, and Sodium Hydrogen Sulphate will be left behind in the dish.

The formation of the two salts is well shown by the following equations, which must be carefully noted :—

$$2NaOH + H_2SO_4 = Na_2SO_4 + 2H_2O.$$
Normal Salt.

$$2NaOH + 2H_2SO_4 = 2NaHSO_4 + 2H_2O.$$
Hydrogen Salt.

CHAPTER XIV

ALKALIMETRY AND ACIDIMETRY

From the preceding experiments we have seen that soluble bases and acids neutralize one another to produce salts, and that the neutralizing point is perfectly definite and can easily and accurately be determined.

Since, according to the Law of Definite Proportions, a given compound always contains the same elements united in the same proportions by weight, it follows that the weights of acid and alkali which neutralize one another to form a given salt are always the same.

Hence it follows that, by performing a neutralizing experiment—usually called a Titration—accurately, in which the strength of one of the solutions, either acid or alkali, is known, the strength of the other can be readily calculated.

This forms a very simple method for determining the strengths of acid or alkaline solutions, unattainable by other means, because most of the acids and alkalis cannot be weighed separately in a pure condition.

As an original solution, some weight of an alkali which can be weighed in a pure state is taken and dissolved in a known volume of water. Such a solution is called a "Standard" solution. From it, by titration, the strength of acid solutions can be obtained and standardized, and from them, by the same process, the strengths of other alkaline solutions can be obtained.

For convenience in calculation, it is usual to make up all volumetric solutions to what is known as "Normal" strength (or some simple fraction of it).

A Normal solution may be defined as one of which 1 litre (1000 c.c.) contains 1 grm. of acid Hydrogen, or its equivalent in grams of any metal.

For instance, since the molecular weight of Hydrochloric acid is 36·5, and that number of grams of it will contain 1 grm. of Hydrogen, the Normal solution of Hydrochloric acid, denoted by N. HCl, will contain 36·5 grms. of Hydrochloric acid per litre of solution.

Similarly, a Normal solution of Caustic Soda (N. NaOH) contains 40 grms. of NaOH per litre, since the molecular weight of NaOH is 40, and that number of grams of it contains 23 grms. of the metal Sodium, which is equivalent to 1 grm. of Hydrogen.

On the other hand, N. H_2SO_4 will contain 49 grms. of acid per litre, since the molecular weight of Sulphuric acid is 98, and that number of grams of it contains 2 grms. of Hydrogen, and therefore the weight of this acid which contains 1 grm. of Hydrogen is 49 grms.

Again, a Normal solution of Lime would contain only 28 grms. of CaO per litre, for the metal Calcium being divalent, its equivalent weight is 20, and this weight in grams is contained in 28 grms. of Lime.

Experiment 90

To prepare a normal solution of Sodium Carbonate.—One of the few alkalis or acids which can be obtained in a pure solid condition, and can consequently be accurately weighed, is anhydrous Sodium Carbonate (Na_2CO_3).

Its molecular weight is 106, and as this contains two

atoms of the monovalent metal Sodium, the weight of it which contains 23 grms.—the equivalent weight of Sodium —is 53 grms.; and therefore N. NaCO solution must contain 53 grms. of Sodium Carbonate per litre.

To prepare this solution, take about 30 grms. of the pure anhydrous salt in a porcelain dish, and dry it, for about half an hour, in a hot-air bath, at a temperature of 150° C. Cool this in a desiccator, and then weigh out into a clock-glass 26·5 grms. of the salt very carefully.

Now place every particle of this in a clean beaker, and dissolve it in about 150 c.c. of distilled water, stirring well and warming slightly if necessary; then transfer the whole of this solution to a flask graduated to hold 500 c.c., wash the beaker out once or twice with distilled water, pouring the washings into the flask, and then fill up with distilled water exactly to the mark on the flask, put in the cork, and shake thoroughly.

This solution is N. Na_2CO_3; it contains 23 grms. of Sodium per litre, or ·023 grm. per c.c.: and 1 c.c. of it is therefore equivalent to ·001 grm. of Acid Hydrogen.

Experiment 91

To find the strength of a solution of Hydrochloric Acid.—To do this it is necessary to take the given solution of Hydrochloric acid, and neutralize it with the N. Na_2CO_3 solution prepared in the last experiment, ascertaining exactly the number of c.c. of acid solution required.

In these acid and alkaline titrations the most accurate results are obtained by placing the acid solution in a burette and adding it to a known quantity of the alkaline solution contained in a beaker.

N.B.—Burettes and pipettes used in Volumetric Analysis must always be rinsed out, before filling, with a few c.c. of the solution to be placed in them: this is because, when put away, they always have a little water adhering to the sides, and this would make weaker any solution which was placed in them. A burette or pipette thus rinsed out is said to be "clean" for that particular solution.

To perform the titration, "clean" a burette with the Hydrochloric acid solution, and fill it up to the zero mark with the same, being especially careful to get rid of all air bubbles at or near the top of the burette; next, "clean" a 10 c.c. pipette, and fill it with the N. Na_2CO_3 solution, and let the contents run out into a beaker, which has previously been rinsed out with distilled water. Then add to the liquid in the beaker one or two drops of strong litmus solution—enough to enable you to see the blue colour—to act as an "indicator" of the point when neutralization is complete.

Now add gradually, a few drops at a time, the acid solution from the burette to the alkali in the beaker: effervescence takes place, owing to the liberation of Carbonic acid gas, and the alkali is gradually neutralized, according to the equation—

$$Na_2CO_3 + 2HCl = 2NaCl + H_2O + CO_2.$$

After a time the Carbonic acid produced begins to change the colour of the litmus, and would mask the turning point of the reaction: this must be obviated by boiling the solu-

tion, when the Carbonic acid gas is driven off, and the litmus is restored to its original blue colour.

Keep on adding the acid gradually till the litmus is turned a red colour which does not become blue on boiling. Note carefully the reading of the burette: this forms a rough indication of the amount of acid required.

Now rinse out the beaker with distilled water, and put in another 10 c.c. of the N. Na_2CO_3 solution, and perform the same operation as before, this time very carefully, so that the colour of the litmus changes when one drop of the acid is added. Note the number of c.c. used, and perform the operation a third time to confirm this result: the last two titrations should show precisely the same volume of acid.

Suppose 7·3 c.c. of the acid solution have been added. Refer again to the equation—

$$\underset{106}{Na_2CO_3} + \underset{2\times 36{\cdot}5}{2HCl} = 2NaCl + H_2O + CO_2.$$

From this we see that 106 grms. of Sodium Carbonate neutralize 73 grms. of Hydrochloric acid, and therefore 53 grms. neutralize 36·5 grms. Therefore 1 c.c. of N. Na_2CO_3 solution containing ·053 grm. of Sodium Carbonate neutralizes ·0365 grm. of Hydrochloric acid. It follows, therefore, that the 10 c.c. of N. Na_2CO_3 used in the experiment will neutralize ·365 grm. of Hydrochloric acid, which weight of acid must therefore be contained in the 7·3 c.c. of its solution which was added from the burette.

Hence 7·3 c.c. of Hydrochloric acid solution contain ·365 grm. of pure Hydrochloric acid, and a litre or 1000 c.c. must contain $\frac{1000}{7{\cdot}3} \times {\cdot}365$, i.e. 50 grms. of Hydrochloric acid.

Instead of using litmus as an indicator in this experiment, Methyl Orange might be used. This substance is a

yellow colour when alkaline and pink when acid, and has the advantage of being unaffected by Carbonic acid, thus obviating the necessity of boiling the solution, and making the operation of titration more rapid. The drawback to its use, however, is that the results of titrations are never so reliable as when Litmus is used.

Experiment 92

To prepare N. HCl.—If the HCl solution analyzed in the last experiment is stronger than the N. Na_2CO_3—that is, if less acid than alkali was needed for the titration—it is quite simple to make up N. HCl from it; but if not, a stronger acid solution must be made up, and its strength ascertained as in the last experiment. Now since Normal solutions contain equivalent quantities of acid Hydrogen and metal per litre, it follows that they neutralize one another in equal quantities by volume, that is, 10 c.c. of N. Na_2CO_3 neutralize 10 c.c. of N. HCl. Hence it follows that if the HCl solution of the last experiment be used, 7·3 c.c. of it must be diluted to 10 c.c. to make it exactly equivalent, bulk for bulk, to the N. Na_2CO_3. So in order to prepare 500 c.c. of the N. HCl, measure out carefully in a measuring-glass 365 c.c. of the HCl solution, place it all in a 500 c.c. flask, rinse out the measuring-glass with distilled water, pouring the rinsings into the flask, and fill up to the mark with distilled water. Shake up vigorously, and this solution should be N. HCl.

Test this by titrating 10 c.c. of the N. Na_2CO_3 with it, when exactly 10 c.c. of the HCl solution should be required.

Experiment 93

To find the strength of a solution of KOH, and to make a solution of N. KOH.—Balance a beaker roughly, and then put into it about 30 grms. of solid KOH; dissolve this in an ordinary flask, which has a capacity of about 300 c.c.; when all is dissolved, shake up thoroughly and allow to cool.

Now take some of the N. HCl prepared in the last experiment in a burette "cleaned" for the solution; take 10 c.c. of the KOH solution in a beaker, and titrate the acid and alkali, using litmus as an indicator. N.B.—Since no CO_2 is given off, no boiling is necessary.

As before, do three titrations, the last two agreeing.

Suppose 15·5 c.c. of N. HCl are needed to neutralize 10 c.c. of the alkaline solution, it follows that the KOH solution is $\frac{15{\cdot}5}{10}$ of Normal.

Now N. KOH contains 39 grms. of K, i.e. 56 grms. of KOH per litre. Therefore the strength of the KOH solution is $\frac{15{\cdot}5}{10} \times 56 = 86{\cdot}8$ grms. per litre.

To make up the solution to normal strength, and therefore equivalent to the acid used, it is clear that if we dilute 10 c.c. of it with water up to 15·5 c.c., then the two solutions will be equivalent to one another.

10 c.c. therefore require 5·5 c.c. of water.

Take 200 c.c. of the KOH solution, add to it the necessary water, viz. 110 c.c., putting both into a clean dry flask; this will be N. KOH. Titrate it against the N. HCl, and see if it is exactly equivalent.

The equation is:—

$$KOH + HCl = KCl + H_2O.$$

Experiment 94

To make up a quantity of N. H_2SO_4.—This is a precisely similar experiment in its working to the last. Make up a strong solution of H_2SO_4, by mixing carefully and slowly 25 c.c. of concentrated H_2SO_4 with 500 c.c. of water in a beaker or flask of thin glass. When cool, place it in a beaker and titrate it against 25 c.c. of the N. KOH prepared in the last experiment.

Suppose 16·5 c.c. of the H_2SO_4 solution are required.

It follows that this solution is $\frac{25}{16{\cdot}5}$ of Normal, and to bring it down to normal strength 16·5 c.c. of it must be made up to 25 c.c. with water, i.e. 8·5 c.c. of water must be added. Take about 20 times these quantities of solution and water, mix them in a clean dry flask, shake up well, and test the resultant liquid against N. KOH or N. NA_2CO_3, to determine if it is exactly equivalent or not.

The equation is:—

$$2KOH + H_2SO_4 = K_2SO_4 + 2H_2O.$$

N. H_2SO_4 contains 49 grms. per litre.

The four normal solutions of Na_2CO_3, HCl KOH, and H_2SO_4 now prepared may, if certified to be accurate, be kept, or placed in a common stock-bottle for future use.

It will be noticed that the accuracy of all these solutions depends upon the accuracy with which the first N. Na_2CO_3 solution was made up: hence the great care needed in its preparation.

Experiment 95

To find the strength of an HCl solution, using Iceland Spar.—This is a very simple but rather tedious method for determining the strength of an acid solution.

A lump of Iceland Spar, about 5–10 grms. in weight, is carefully weighed out; it is then placed in a beaker containing 25 c.c. of the given acid solution. Effervescence takes place, and the acid is gradually "killed" or neutralized by the Calcium Carbonate. When no more effervescence occurs, even on warming, take out the lump of Spar, dry it with filter paper, and weigh it again carefully. Note its loss in weight.

The equation is:—

$$\underset{100}{CaCO_3} + \underset{73}{2HCl} = CaCl_2 + CO_2 + H_2O.$$

We see that 100 grms. of Chalk neutralize 73 grms. of HCl. Therefore for every gram of Iceland Spar dissolved ·73 grm. of HCl must have been present: hence the strength of the acid solution can be readily ascertained.

Experiment 96

To determine the percentage of Ammonia in any Ammonium compound.—(1) This determination depends for its working on the fact that when any Ammonium compound is boiled with a solution of Caustic Potash, Ammonia gas is driven off, and the alkaline properties of the Caustic Potash partly disappear.

This may be made clearer by the following example, in which Ammonium Chloride is used. The equation is:—

$$\underset{56}{NH_4Cl + KOH} = HCl + \underset{17}{NH_3} + H_2O.$$

It is clear that the Caustic Potash, being changed into Potassium Chloride, loses its alkaline character, and is neutralized: also it appears from the equation that 56 grms. of KOH will drive out 17 grms. of Ammonia from any Ammonium salt. Consequently 1 c.c. of N. KOH solution, which contains ·056 grm. of KOH, will drive out ·017 grm. of Ammonia.

To perform the experiment, 1 grm. of the Ammonium salt must be weighed out and placed in a flask, 50 c.c. of N. KOH be added, and the contents of the flask vigorously boiled, till the steam given off has no action on either red litmus or turmeric paper, showing that all the Ammonia has been driven off.

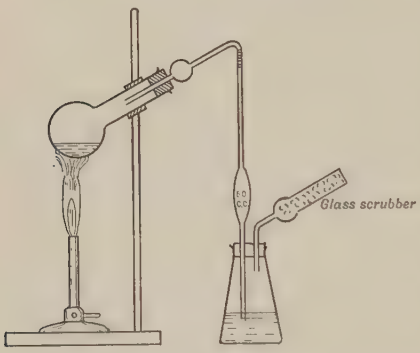
N.B.—During the boiling the flask must be held in a slanting position to avoid loss by spurting.

When the boiling is finished, titrate the solution in the flask with N. H_2SO_4, to find out how much of the 50 c.c. N. KOH is left unacted upon by the Ammonium salt.

It is clear that if the titration requires 20 c.c. of N. H_2SO_4,

20 c.c. of the N. KOH must have been added in excess, and hence 30 c.c. has been used up. This contains enough KOH to liberate $30 \times \cdot 017$ grms. of Ammonia, which must have been contained in the original gram of Ammonium salt: hence the percentage of Ammonia present in the salt can be readily calculated.

(2) This method consists in taking 1 grm. of the Ammonium salt, adding excess of strong KOH solution, and boiling off all the Ammonia. The Ammonia is led into a known volume of normal acid, part of which it neutralizes, and the quantity left unneutralized is determined by titration with N. KOH.



The apparatus used is shown in the diagram. One gram of the Ammonium salt is weighed out, and dissolved in about 25 c.c. of water: this is all placed in the flask, and half a stick of KOH is added. The delivery-tube is fitted first with a bulb-tube, to prevent any spurting over, and this is joined to a 50 c.c. pipette which passes through the cork of a flask containing 50 c.c. of the N. H_2SO_4 previously poured in through the drying-tube, which is filled with broken pieces of glass. Now boil the mixture in the flask (avoid bumping by putting in pieces of Platinum wire): the Ammonia passes out through the pipette, which dips just below the surface of the N. H_2SO_4, and if any is not absorbed by the acid in the flask, it does not get past that left on the

pieces of glass in the drying-tube. "Sucking back" into the flask is prevented by the "bulge" of the pipette.

After boiling steadily for some time—say ten minutes—disconnect the apparatus, wash the acid on the broken glass through into the flask with distilled water, and then titrate the acid with N. KOH, using litmus as an indicator.

Suppose 27 c.c. of N. KOH were required, indicating that 27 c.c. of the normal acid was still unneutralized, and that only 23 c.c. had been neutralized by the Ammonia.

Now every c.c. contains ·049 grm. of H_2SO_4, and from the equation

$$\underset{98}{H_2SO_4} + 2\underset{34}{NH_3} = (NH_4)_2SO_4$$

it is clear that this weight of H_2SO_4 will neutralize ·017 grm. of Ammonia. Therefore the original salt must have contained $23 \times ·017$ grms. of Ammonia in 1 grm.: hence the percentage is easily calculated.

CHAPTER XV

SOME SIMPLE VOLUMETRIC ESTIMATIONS

Use of processes of Oxidation in Volumetric Analysis. — Standard solutions of oxidizing agents are used very largely in Volumetric Analysis, particularly for estimating the quantity of Iron present in any given solution.

The two oxidizers most generally used are Potassium Bichromate and Potassium Permanganate.

Use of Potassium Bichromate.—This substance, used in conjunction with Sulphuric acid, possesses great oxidizing power, and readily turns Ferrous compounds into Ferric.

The reaction by which this is accomplished is seen in the equation—

$$\underset{294}{K_2Cr_2O_7} + 6FeSO_4 + 7H_2SO_4 = K_2SO_4 + Cr_2(SO_4)_3 + 3Fe(SO_4)_3 + 7H_2O.$$

This action proceeds in a similar manner if the Iron be present as a Chloride or any other soluble salt, instead of a Sulphate.

It will be seen from the equation that 294 grms. $K_2Cr_2O_7$ are capable of changing 336 grms. of Iron from the ferrous to the ferric state, and the standard solution of $K_2Cr_2O_7$ is made up accordingly (see below).

The end of the reaction is marked by the disappearance of the last trace of ferrous salt from the solution. This cannot be seen in the solution itself, nor is any reliable indicator available for use in the solution.

Use is made of a very delicate reaction ferrous salts have with Potassium Ferricyanide, a concentration of 1 part in 1,000,000 giving a distinct blue colour to this reagent. Drops of the Iron liquid are taken out from time to time, and tested with the Potassium Ferricyanide, till no trace of colour is seen: this marks the entire change from "ferrous" to "ferric."

Experiment 97

To prepare a standard solution of Potassium Bichromate.—From the equation given above it is seen that 294 grms. of $K_2Cr_2O_7$ will oxidize 336 grms. of ferrous Iron to ferric, so that 49 grms. will oxidize 56 grms. (i.e. the atomic weight in grams) of Iron.

A solution, therefore, if made up to contain 4·9 grms. of $K_2Cr_2O_7$ per litre, is of such strength that 1 c.c. of it will oxidize ·0056 grm. of Iron.

To prepare it, melt 7 or 8 grms. of $K_2Cr_2O_7$ in a porcelain dish to drive off all moisture, allow to cool, break up the solidified salt, and weigh out accurately 4·9 grms. of it. Dissolve this in a small quantity of distilled water in a beaker, and transfer the whole to a litre flask, fill up to the mark with distilled water, and shake well.

N.B.—This solution keeps well in a stoppered bottle.

It is best next to confirm the oxidizing power of the standard solution of $K_2Cr_2O_7$ by titrating it against a known weight of pure Iron in solution.

Experiment 98

To prepare a solution of Iron of known strength.—Weigh out accurately 1 grm. of pure Iron wire, place it in a flask, add 100 c.c. of dilute Sulphuric acid (1 to 8), and fit the flask with a Bunsen valve.

This valve consists of a piece of thin rubber tubing about 3 in. long, fitted on to the short delivery-tube leading from the flask (which should have a rubber cork). The open end of the tube is closed with a piece of glass rod, and a slit about half an inch long is made with a sharp knife longitudinally in the rubber tubing: this allows gas or steam to escape from the flask, but, if the edges are cleanly cut, will not permit air to come in from the outside, since the slit will be closed by the atmospheric pressure.

Thus no Oxygen from the air can get at the ferrous solution while it is in a hot condition and very susceptible to oxidation; when cool it does not oxidize so easily.

While the iron wire is dissolving, boil about 300 c.c. of distilled water in a flask to expel all dissolved Oxygen, and allow to cool.

When all the iron is dissolved and the contents of the flask quite cool, quickly transfer the whole of the contents to a 250 c.c. flask, rinse out the flask which held the Iron solution with the boiled distilled water, pour the rinsings into the 250 c.c. flask, and fill up to the mark. This solution is clearly one which contains ·1 grm. of Iron in every 25 c.c. N.B.—It will not keep more than 24 hours.

Experiment 99

To standardize the Potassium Bichromate solution.—First make up a few c.c. of a solution of Potassium Ferricyanide, and place a dozen or so drops of it on a white china plate: these drops serve as an indicator.

Then take 25 c.c. of the freshly prepared solution of Iron (which is in the ferrous state) in a beaker, and add to it gradually the Bichromate solution from a burette. Stir the mixture very thoroughly with a light glass stirring-rod (made by closing up the ends of a piece of glass tube), take out one drop on the end of the stirrer, and place it in one of the drops of Ferricyanide on the white plate. If a blue colour is seen, it follows that there must be still some Iron present in a ferrous condition—unoxidized.

Continue to add the Bichromate, stirring well after each addition, till no trace of the blue colour is seen in the indicator, and only a reddish coloration appears: this should be produced by the addition of only one drop of Bichromate solution since the last trace of blue colour was seen.

Now note the volume of Bichromate added.

Since 25 c.c. of the Iron solution contain ·1 grm. of Iron, it follows that, if x c.c. of Bichromate solution have been added, 1 c.c. of this solution must be equivalent to $\frac{1}{10x}$ grm. of Iron. $\frac{1}{10x}$ should be equal to ·0056, if the solutions have been accurately prepared.

Experiment 100

Titration of the standard Potassium Bichromate by means of a Ferrous salt.—A salt which contains Iron in the ferrous state, and which can be obtained in a very pure condition, is Ferrous Ammonium Sulphate ($FeAm_2(SO_4)_2 6H_2O$). It is also remarkable in containing exactly one-seventh of its weight of Iron. If, therefore, 2·8 grms. of this salt be weighed out, and dissolved in 100 c.c. of cold water, free from air and containing a little dilute Sulphuric acid, then 25 c.c. of this solution will contain ·1 grm. of Iron, and if titrated against the Bichromate solution, as in Experiment 98, additional confirmation of its strength can be obtained.

Having thus accurately standardized the Bichromate solution, it can be used for determining the quantity of Iron present in any solid or solution containing it.

Experiment 101

To determine the percentage of Iron in crystals of Ferrous Sulphate.—Weigh out 5 grms. of Ferrous Sulphate crystals, dissolve them in a little air-free water, adding a few c.c. of strong Sulphuric acid; transfer the solution to a 250 c.c. flask, wash out the beaker into the flask with air-free water, and fill up to the mark.

25 c.c. of this solution is taken out, and titrated as described in Experiment 99, and from the result of the titration the percentage of Iron can readily be calculated.

If Iron is present in the ferric condition, it must be reduced to the ferrous state before titration can be of any value.

Experiment 102

To determine the percentage of Iron in Iron Alum ($K_2Fe_2(SO_4)_4 24H_2O$).—Weigh out 10 grms. of Iron Alum, and dissolve it in water; add a little Sulphuric acid, and make up the solution to 250 c.c.

The reduction of the ferric Iron may be brought about by two methods.

(1) Reduction by means of Zinc.

Take 100 c.c. of the Iron Alum solution in a flask fitted with a Bunsen valve (see Experiment 97). Add 20 c.c. of strong HCl and about 5 grms. of pure granulated Zinc, free from Iron.

The nascent Hydrogen produced by the action of the acid on the Zinc will reduce the ferric to ferrous Iron.

When all the Zinc has dissolved (heat may be applied if necessary) and the solution cooled, it is transferred to a 250 c.c. flask, which is filled up to the mark with air-free water.

The quantity of Iron present in this solution, and hence in the original Iron Alum, can be readily determined by the titration of 50 c.c. of it with Potassium Bichromate.

This titration must be performed at once, as the liquid does not keep.

(2) Reduction by means of Stannous Chloride solution.

25 c c. of the Iron Alum solution are taken in a beaker, 2 or 3 c.c. of strong HCl are added, and the liquid

heated to boiling. Clear freshly made Stannous Chloride solution is then added drop by drop from a pipette till the yellow colour of the solution just disappears: this indicates that the Iron has all been reduced to the ferrous state by the Stannous Chloride according to the equation—

$$2FeCl_3 + SnCl_2 = 2FeCl_2 + SnCl_4.$$

The excess of Stannous Chloride solution is got rid of by adding Mercuric Chloride solution, which must produce turbidity, showing that excess of Stannous Chloride was present. The turbid liquid is allowed to cool a little, and then titrated with Potassium Bichromate in the usual way.

Use of Potassium Permanganate

Potassium Permanganate is another oxidizing agent used in the determination of Iron in solutions containing it. It cannot, however, be used in the presence of organic matter; also it is untrustworthy in the presence of HCl.

Unfortunately also its solution becomes gradually weaker if exposed to light, owing to the decomposition of the salt. It should be kept in a dark blue glass bottle, and stored in a dark cool place: even then it must be standardized at frequent intervals.

Experiment 103

Preparation and standardization of Potassium Permanganate solution—About 5 grms. of pure, re-crystallized Potassium Permanganate should be dissolved in a litre of cold water, and at once transferred to the dark blue bottle in which it is to be kept.

It is standardized by using a cold solution of ferrous Ammonium Sulphate, containing some free Sulphuric acid.

The following reaction takes place (using Ferrous Sulphate instead of Ferrous Ammonium Sulphate in the equation):—

$$K_2Mn_2O_8 + 10FeSO_4 + 8H_2SO_4 = 5Fe_2(SO_4)_3 + K_2SO_4 + 2MnSO_4 + 8H_2O.$$

A solution of Ferrous Ammonium Sulphate, containing ·1 grm. of Iron in 25 c.c., is freshly made up as in Experiment 99.

25 c.c. are taken out, a few c.c. of dilute Sulphuric acid are added to this, and the liquid is titrated with the Potassium Permanganate solution, which must be contained in a burette with a glass stop-cock, as rubber would reduce the permanganate. As soon as all the Iron is oxidized, a faint pink colour—due to very slight excess of the permanganate—is seen, and this marks the turning point of the reaction.

Two titrations should be made—they should not differ by more than one-tenth of a c.c.—and from this result the weight of Iron oxidized by 1 c.c. of the Potassium Permanganate solution can be readily obtained.

The Potassium Permanganate solution may be used for all determinations of Iron, whether in the ferrous or ferric state, provided always that HCl is not present.

Hence reduction preceding oxidation by Potassium Permanganate cannot be made with Stannous Chloride, but must always be done with Zinc and Sulphuric acid.

Titration of Potassium Permanganate Solution by Means of Oxalic Acid

When Potassium Permanganate solution is added to a hot solution of Oxalic acid in the presence of Sulphuric acid, the following reaction takes place:—

$$5(COOH)_2 + K_2Mn_2O_8 + 3H_2SO_4 = 10CO_2 + K_2SO_4 + 2MnSO_4 + 3H_2O.$$

Hence it will be seen that 5 molecules of Oxalic acid are oxidized by 1 molecule of Potassium Permanganate.

Experiment 104

To prepare a solution of Oxalic acid and titrate it with the Potassium Permanganate solution.—Oxalic acid contains 2 molecules of water of crystallization, and therefore the molecular weight of the crystalline salt is 126.

Dissolve, therefore, 1·575 grms. of the pure recrystallized acid in water and make up the solution to 250 c.c.

Then 1 c.c. of this solution contains ·0063 grm. of the crystalline Oxalic acid, and will be oxidized by ·00314 grm. of Potassium Permanganate.

The titration is performed as in the case of an Iron salt, always remembering that the solution must be hot.

The strength of a solution of a soluble Oxalate may also be determined by the Potassium Permanganate solution in a similar manner.

Estimation of Silver by Standard Sodium Chloride Solution.—When a solution of Silver Nitrate is mixed with a solution of Sodium Chloride, the Silver is precipitated as white insoluble Chloride of Silver, according to the following equation :—

$$AgNO_3 + NaCl = AgCl + NaNO_3.$$

A similar reaction occurs with any soluble Silver salt.

It is a simple matter, therefore, to estimate the amount of Silver in any solution by exactly precipitating a known volume of standard Sodium Chloride solution by means of it.

As an indicator of complete precipitation, a few drops of Potassium Chromate are added to the Salt solution; this forms a dark red precipitate of Silver Chromate as soon as the whole of the Chloride has been precipitated.

N.B.—This indicator is useless in the presence of free acid, such as Nitric acid.

Experiment 105

To prepare a deci-molecular solution of Sodium Chloride, and to use it for determining the strength of a Silver solution.—From the above equation it is seen that 5·85 grms. of NaCl exactly precipitate ·108 grm. of Silver.

Weigh out accurately 5·85 grms. of pure NaCl, dissolve it in water, and make the solution up to 1 litre.

Then each c.c. of this solution contains ·00585 grm. of NaCl, and is equivalent to ·0108 grm. of Silver.

In the actual titration, 10 c.c. of the NaCl solution are taken in a porcelain dish, diluted with a little distilled water, and three or four drops of Potassium Chromate solution are added with constant stirring of the liquid in the dish, till one drop produces a faint permanent red tint.

This marks the end of the reaction. Then it is clear that the volume of liquid added from the burette must contain ·108 grm. of Silver.

Experiment 106

To determine the percentage of Silver in a silver coin.—Weigh out about ·5 grm. of the coin, and dissolve it in dilute Nitric acid, making the liquid, when the solution is complete, up to 100 c.c.

25 c.c. of this acid solution is taken in a stoppered bottle, and to it the deci-molecular solution of NaCl is added. N.B.—During the titration the bottle must be wrapped up in a dark cloth to prevent the access of light.

After every addition of NaCl solution the bottle is shaken vigorously, till the precipitate of Silver Chloride is clotted together. It is then allowed to settle till the supernatant liquid is clear. This procedure is continued till the last drop of NaCl solution fails to produce turbidity in the supernatant liquid. Then, since each c.c. of NaCl is equivalent to ·0108 grm. of Silver, the percentage of Silver in the coin can readily be calculated.

Experiment 107

To determine the percentage of Chlorine in a soluble Chloride.—Since we are able to determine the percentage of Silver by means of a solution of NaCl, it is clear that we can readily determine the percentage of Chlorine by means of a solution of Silver Nitrate.

The solution is made up in deci-molecular strength, i.e. 27 grms. per litre: only 100 c.c. should be made up at once, and it must be kept in black bottles to protect it from the light.

From the equation

$$AgNO_3 + NaCl = AgCl + NaNO_3$$

it is seen that 270 grms. of Silver Nitrate are equivalent to 35·5 grms. of Chlorine, and therefore 1 c.c. of this solution is equal to ·00355 grm. of Chlorine.

One gram of the soluble Chloride is weighed out and dissolved in 100 c.c. of water, and 10 c.c. of this solution is titrated with the Silver Nitrate solution as in Experiment 104, using Potassium Chromate as an indicator. From the above relation the percentage of Chlorine in the Chloride may be readily calculated.

CHAPTER XVI

ANALYSIS OF A SIMPLE SALT

As we have previously learnt, a salt is formed by the neutralization of an acid by a base, and therefore may be said to contain an acid and a base, and the analysis of a simple salt consists in the determination of what the acid and base are.

Such an analysis consists in the application of a series of tests or experiments to the salt—or its solution—the careful observation of all changes which take place when the salt is undergoing these tests, and the drawing of inferences as to the presence or absence of certain bases or acids from the observations made.

These tests must be applied in a regular order or sequence; otherwise the inferences drawn from them are valueless.

(1) A series of Preliminary Tests is made for the base.

(2) The solution is then tested by a series of Group Tests, by which the identity of the base should be firmly established.

(3) At least two Confirmatory Tests for the base should then be done.

(4) A series of Preliminary Tests for the acid is now made, which should practically decide which acid is present.

(5) At least two Confirmatory Tests for the acid should then be done.

There are one or two experiments peculiar to analytical work, which must first be thoroughly familiarized.

(*a*) Flame Reactions.

Fuse a piece of platinum wire about 2 in. long into the end of a short piece of glass tubing; dip this into a mixture of a small quantity of the solid to be analysed and one or two drops of strong Hydrochloric acid previously made on a watch-glass, and hold the wire in a Bunsen flame.

Certain bases give distinctive colours to the flame, and by these they may be recognized.

(*b*) Borax Bead Reactions.

Bend the end of the platinum wire into a small loop, dip it when hot into some powdered Borax, and heat in the flame. On repeating this several times, a clear glassy mass is obtained on the end of the wire—this is a Borax Bead. On dipping this bead into a very small fragment of the unknown salt, and then heating it again in the blow-pipe flame, colours are imparted to the bead by certain bases, and by these they may be recognized.

These colours sometimes vary according as the bead is heated in the inner or the outer flame.

(*c*) Reductions on Charcoal.

Many of the salts of the heavier metals are reduced to the metal itself by being heated in a small cavity on a piece of wood charcoal in the inner or reducing flame.

This reduction is often very much accelerated by adding Sodium Carbonate to the salt before heating. This forms a Carbonate of the metal present in the salt, which, being

heated, changes to Oxide, and this Oxide is usually more readily reduced by the charcoal than the original salt.

Potassium Cyanide is another solid used to promote rapid reduction on charcoal.

(*d*) Precipitation.

When two clear and transparent solutions are mixed together, they often become more or less turbid or opaque, owing to the formation of a solid substance in the liquid. The solid thus produced is called a Precipitate; these precipitates are variously coloured, and being recognized, reveal the presence of various bases or acids.

Any substance which is used for producing such a precipitate is called a Reagent.

In analytical work it is best to take notes in three columns, labelled respectively, "Experiment," "Observation," "Inference." Each experiment should be noted down immediately on completion, with the changes observed and the inferences drawn from them.

N.B.—If no apparent change takes place, it should still be noted down, as important deductions may often be drawn from this lack of action.

A specimen of notes, showing how a complete analysis should be entered in the notebook, is given on page 130.

Before proceeding to apply tests to the substance to be analysed, its appearance should be carefully noted down.

Observe whether it is crystalline or amorphous; note its smell and colour. These often afford valuable indications.

The following are some of the commoner coloured compounds:—

Blue—Copper salts.

Green—Ferrous salts; also salts of Nickel and Chromium.

Yellow—HgO, As_2S_3, PbI_2, and all Chromates.

Red—HgO, HgI_2, HgS, Pb_3O_4, Sb_2S_3, and orange-red Bichromates.

Pink—Salts of Mn and Co.

Brown—Fe_2O_3, PbO_2; PbO, Bi_2O_3, CdO are yellowish brown.

N.B.—It is inadvisable to taste the substance for analysis, many salts being poisonous, even in very small quantities.

PRELIMINARY EXAMINATION FOR THE BASE

Experiment	Observation	Inference
1. Heat a small quantity of the solid substance in a test-tube.	A. **The substance does not change.**	Absence of volatile substances and of water.
	B. **The substance changes.**	
	1. **It changes colour.**	
	Hot / **Cold**	
	Yellow. / White.	ZnO.
	Yellow-brown. / Yellow.	PbO.
	„ / White.	SnO_2, Bi_2O_3.
	Black. / Brown.	Fe_2O_3.
	Dark red. / Bright red.	Pb_3O_4, HgO.
	From green to black.	$CuCO_3$, $Cu(NO_3)_2$.
	2. **The substance chars.**	Presence of Organic acid.
	3. **It sublimes.**	
	White sublimate.	NH_4, $HgCl_2$, As_2O_3.
	Yellow „	As_2S_3, HgI_2.
Confirmatory Tests.		
(1) Mix the substance with three times its weight of Na_2CO_3, and heat in a small test-tube.	Grey mirror of small globules formed on sides of tube.	Hg.
	Black shining mirror formed.	As.
(2) Add a little KOH solution to solid, and heat.	NH_3 gas given off, recognized by smell and alkaline reaction.	NH_4.
	4. **It gives off water.**	Presence of water of crystallization or Hydrate.
Test the water drops with Litmus.		
	(*a*) The water is alkaline.	NH_4.
	(*b*) „ „ acid.	Sulphate, Nitrate, or Chloride.

Experiment	Observation	Inference
	5. A gas is given off.	
	(*a*) Gas has no smell.	
Confirmatory Tests.		
(1) Place a glowing splinter in mouth of test-tube.	Splinter rekindled.	O, from Chlorate, Nitrate, or Peroxide.
(2) Bubble the gas into Lime-water.	Produces milkiness.	CO_2, from a Carbonate or Oxalate.
(3) Apply a light to mouth of tube.	Gas burns with a blue flame.	CO, from an Oxalate.
	(*b*) Gas has a smell.	
Confirmatory Tests.		
(1) Test with wet Litmus paper.	Fumes are reddish and acid.	NO_2, from a Nitrate or Nitrite.
(2) Test with paper dipped in solution of $K_2Cr_2O_7$.	$K_2Cr_2O_7$ is turned green.	SO_2, from some Sulphur compound, or from free Sulphur.
2. Place a little of the substance in a watch-glass, moisten with HCl, dip into the mixture a clean platinum wire, and hold it in the flame.	The flame is coloured—	
	1. Intense yellow.	Na.
	2. Pale lilac.	K.
	3. Yellowish green.	Ba.
	4. Bright green.	Cu or a Borate.
	5. Crimson.	Sr.
	6. Yellowish red.	Ca.
	7. Livid blue.	Cu, As, Sb, Pb.
3. Heat a little of the powdered substance in a cavity made in a piece of charcoal, before the blow-pipe.	(*a*) The substance deflagrates or causes the charcoal to burn rapidly.	Chlorate or Nitrate.
	(*b*) The substance fuses easily, and is absorbed by the charcoal, or forms a liquid bead.	Salt of an alkali metal.

Experiment	Observation		Inference
	(*c*) An infusible residue is left on the charcoal.		
	1. The residue is white and luminous when hot.		Ba, Sr, Ca, Mg, Al, Zn, as Oxides.
Confirmatory Test.			
Put 3 or 4 drops of $Co(NO_3)_2$ solution on the white residue, and heat again.	A blue residue.		Al, or a phosphate.
	A pink residue.		Mg.
	A green residue.		Zn.
	2. The residue is coloured.		
	Colour of Bead.		
	O.F.	I.F.	
Confirmatory Test	Green, hot. Blue, cold.	Colourless or Red, cold.	Cu.
Heat a very small quantity of the solid in a Borax bead, first in the outer, then in the inner flame.	Blue.	Blue.	Co.
	Violet, hot. Yellow, cold.	Opaque.	Ni.
	Red-brown, hot. Yellow, cold.	Olive-green.	Fe.
	Amethyst.	Colourless.	Mn
	3. The residue is coloured, and metallic globules are seen.		
	Bead.	Incrustation.	
Confirmatory Test.	Bright white.	None.	Ag.
	Red scales.	None.	Cu.
Heat some of the substance again with Na_2CO_3 or fusion mixture on charcoal.	Grey magnetic powder.	None.	Fe, Ni, Co, Mn.
	White malleable, marks paper.	Yellow.	Pb.
	White malleable, does not mark paper.	Very little.	Sn.
	White brittle.	Yellow.	Bi.
	White brittle, white fumes.	White.	Sb.
	None.	Reddish brown	Cd.
	None.	White, with smell of garlic.	As

Group Tests

These tests consist of applying to the solution of the substance a series of reagents in a certain specified order. These reagents cause Precipitates to form, consisting of insoluble salts of the Base present, the Bases being separated, owing to the insolubility of certain of their salts, into various groups, and the Precipitates in each group being separated either by their colour or by the action of various reagents on them.

Process of Solution

The substance must first be obtained in solution as follows :—

(1) Place as much of the solid as will cover a threepenny-piece in a clean test-tube, add distilled water, and boil.
If insoluble—

(2) Do the same thing with dilute HCl.
If insoluble—

(3) Try strong HCl.
If insoluble—

(4) Try dilute and then strong HNO_3.
If insoluble—

(5) Mix test-tubes (3) and (4), which gives Aqua Regia. If you have to use Aqua Regia, evaporate to dryness with excess of HCl, and dissolve the residue in warm water.
If still insoluble—

(6) Mix the substance with four or five times its weight of Fusion Mixture, and heat strongly in a crucible for ten minutes, till fused. When cold, boil the whole in water till the mass breaks up, and filter.

The Filtrate now contains the Acid, and the residue contains the Base. Dissolve it in dilute HCl.

As soon as a solvent is found, dissolve as much of the solid as will cover a shilling in a boiling-tube, half full of the solvent ; cool if necessary, and use this solution for the "Group Tests." It is called the "Original Solution."

Group I.

Experiment	Observation	Inference
Add dilute HCl to solution.	White ppt.	Ag, Pb, Hg (-ous).
To distinguish, add NH_4OH to ppt.	Ppt. dissolves.	Ag.
	Ppt. blackens.	Hg (-ous).
	No change.	Pb.

Group II.

Experiment	Observation	Inference
Pass H_2S gas through the solution for some time.	Dark-coloured ppt.	Pb, Hg (-ic) Cu, Bi, Sn.
	Yellow ppt.	Cd, As.
	Orange ppt.	Sb.
To distinguish dark-coloured ppts.		
(*a*) Boil part of ppt. with KOH.	Ppt. dissolves.	Sn.
(*b*) If insoluble in KOH, boil rest of ppt. with HNO_3.	No change.	Hg (-ic).
	Ppt. turns white.	Pb.
	Ppt. dissolves, giving blue solution.	Cu.
	Ppt. dissolves, giving colourless solution.	Bi.
To distinguish yellow ppts., boil with KOH.	Ppt. dissolves.	As.
	Ppt. insoluble.	Cd.

Group IIIa.

Take a fresh portion of original solution. Add first NH_4Cl in excess, then NH_4OH.	White ppt. turning dingy green and then rusty red in the air.	Fe (-ous).
	Fox-red ppt.	Fe (-ic).
	Green ppt.	Cr.
	White gelatinous ppt.	Al.

Group IIIb.

To the alkaline liquid add $(NH_4)_2S$.	Black ppt.	Ni, Co
	White ppt.	Zn.
	Flesh-coloured ppt.	Mn.
To distinguish the black ppts., boil with excess of $(NH_4)_2S$ and filter.	Brown filtrate.	Ni.
	Yellow filtrate.	Co.

Group IV.

Experiment	Observation	Inference
To the same liquid add $(NH_4)_2CO_3$.	White ppt.	Ba, Sr, Ca.
To distinguish, add $CaSO_4$ to the original solution.	White ppt. at once.	Ba.
	White ppt. on standing and boiling.	Sr.
	No ppt.	Ca.

Group V.

To the same liquid add Na_2HPO_4.	White ppt.	Mg.

Metals which may be present if no ppts. have been obtained so far are (NH_4), K, Na.

(1) Boil original solution with KOH.	NH_3 gas given off, recognized by pungent smell, and alkaline reaction.	NH_4.
(2) Test original substance on Platinum wire in the flame.	Lilac flame.	K.
	Intense yellow flame.	Na.

CONFIRMATORY TESTS FOR THE BASE.

N.B.—These tests must be made with the original solution, unless otherwise specified.

Silver.

1. Add K_2CrO_4 : deep red-brown ppt. of Ag_2CrO_4.
2. Add a few drops of dilute NH_4OH: brown ppt. of AgOH soluble in excess of the reagent.
3. Heat the solid substance on charcoal before the blow-pipe with a little Na_2CO_3 : bright malleable bead, which does not mark paper ; no incrustation.

Lead.

1. Add H_2S : black ppt. of PbS, turned white ($PbSO_4$) by boiling with HNO_3.
2. Add KI : yellow ppt. of PbI_2 soluble on boiling, but appearing again as beautiful golden crystals on cooling.
3. Add K_2CrO_4 : yellow ppt. of $PbCrO_4$.
4. Heat the solid on charcoal before the blow-pipe with a little Na_2CO_3 : malleable bead which marks paper ; yellow incrustation.

Mercury (Mercurosum).

1. Add NH_4OH : black ppt. of HgOH.
2. Add KI : green ppt of HgI.
3. Add a few drops of HNO_3, then dip into the solution a clean strip of Copper : a grey film of Hg is formed on the Copper.
4. Mix a little of the solid with Na_2CO_3, and heat in a small dry test-tube : a mirror of Hg is formed on the sides of the tube.

Mercury (Mercuricum).

1. Add KOH : yellow ppt. of HgO.
2. Add KI : scarlet ppt. of HgI_2.

3 and 4 as for Mercury (Mercurosum).

Copper.

1. Add a little dilute NH_4OH : light blue ppt. of $Cu(OH)_2$ soluble in excess of NH_4OH, forming a splendid dark blue solution.
2. Add a little $K_4Fe(CN)_6$: a chocolate-coloured ppt. of $Cu_2Fe(CN)_6$.
3. Add a few drops of H_2SO_4, then place in the solution a piece of clean iron or steel : a red deposit of Cu is formed on the iron.
4. Test a small fragment of the solid in a Borax bead : reddish in the inner, blue in the outer flame.

Bismuth.

1. Add NH_4OH : white ppt. of $Bi(OH)_3$.
2. Filter ppt. from (1), dissolve ppt. in a few drops of strong HCl, and add the solution to a large quantity of water in a beaker : a milkiness is produced, due to a ppt. of BiOCl.
3. Heat the solid on charcoal before the blow-pipe with a little fusion mixture : brittle bead ; yellow incrustation.

Cadmium.

1. Add H_2S : bright yellow ppt. insoluble in boiling KOH.
2. Heat the solid on charcoal before the blow-pipe with a little Na_2CO_3 : a brown incrustation is formed.

Arsenic.

1. *Reinsch's Test.*—Add a little HCl and a few pieces of clean Cu, and boil. A dark grey film of Cu_3As_2 is deposited on the Cu. Dry the Cu strips and heat them in a dry test-tube : a white sublimate of As_2O_5 is formed on the sides of the tube.

2. *Marsh's Test.*—Set up an apparatus for preparing pure Hydrogen from *pure* Zn and HCl; add a little of the As solution, and light the jet of H; allow it to impinge on a cold piece of porcelain; a black stain of As is formed on the porcelain; this stain is soluble in a solution of Bleaching Powder.
3. Mix the solid with Na_2CO_3 and heat in a small dry test-tube; a black deposit of As is formed on the sides of the tube.

Antimony.

1. *Marsh's Test.* (See "Arsenic.")—A black stain of Sb is formed on the porcelain, but it is insoluble in a solution of Bleaching Powder.
2. Place a small piece of Platinum foil and a piece of Zn in a porcelain dish, add the Sb solution acidified with HCl: a black stain of Sb is formed on the Platinum.
3. Add the solution, acidified with HCl, to a large quantity of water: a white ppt. of SbOCl is formed.

Tin.

1. Acidify the solution with HCl, and add to it a small piece of Zn: a grey deposit of Sn is formed on the Zn.
2. Add $HgCl_2$: a white ppt. of HgCl is formed with Stannous salts, but no ppt. with Stannic salts.
3. Heat the solid on charcoal before the blow-pipe with a little fusion mixture: a white malleable bead is formed, which does not mark paper.

Iron.

	Reagent	*Ferrous salts*	*Ferric salts*
1.	NH_4OH.	Dirty-green ppt.	Fox-red ppt.
2.	$K_3Fe(CN)_6$.	Dark blue ppt.	Brown coloration.
3.	$K_4Fe(CN)_6$.	Light blue ppt.	Dark blue ppt.
4.	KCNS.	—	Blood-red coloration.

5. Borax bead is green in the reducing flame and yellow in the oxidizing flame for both Ferrous and Ferric salts.

Aluminium.

1. Add NaOH gradually: a white gelatinous ppt. of $Al(OH)_3$, soluble in excess.
2. Add $(NH_4)_2S$: a white gelatinous ppt. of the same.
3. Heat the solid on charcoal before the blow-pipe: white infusible residue; add $Co(NO_3)_2$ and heat again: a blue residue is formed.

Chromium.

1. Add NaOH gradually : a bluish-green ppt. of $Cr(OH)_3$, soluble in excess.
2. Fuse some of the solid on Platinum foil with fusion mixture : a yellow mass of Na_2CrO_4 is formed, which if dissolved in water gives a yellow ppt. with Lead acetate.
3. Borax bead is green in both flames.

Zinc.

1. Add NaOH gradually : a white ppt. of $Zn(OH)_2$ soluble in excess.
2. Add $K_4Fe(CN)_6$: a white ppt. of Zinc ferro-cyanide.
3. Heat the solid on charcoal : a white residue is left, which when moistened with $Co(NO_3)_2$ and heated again turns green.

Manganese.

1. Add NaOH : a white ppt. of $Mn(OH)_2$.
2. Fuse the solid on platinum foil with fusion mixture : a bluish green mass of Sodium Manganate is left.
3. Borax bead is amethyst in the oxidizing flame, colourless in the reducing.

Nickel.

1. Add NaOH : a pale green ppt. of $Ni(OH)_2$.
2. Add KCN : a greenish yellow ppt. of $Ni(CN)_2$, soluble in excess.
3. Borax bead is brownish violet in the oxidizing flame, grey in the reducing.

Cobalt.

1. Add NaOH gradually : a blue ppt. of a basic salt is formed first, which changes to pink $Co(OH)_2$ on heating.
2. Add KCN : a reddish brown ppt. of $Co(CN)_2$, soluble in excess to a brown solution.
3. Borax bead is blue in both flames.

Barium.

1. Add $CaSO_4$: immediate ppt. of white $BaSO_4$.
2. Add K_2CrO_4 : a yellow ppt. insoluble in Acetic acid.
3. Flame test : apple-green colour.

Strontium.

1. Add $CaSO_4$: a white ppt. of $SrSO_4$, after a time, or immediately on boiling.
2. Add $(NH_4)_2C_2O_4$: a white ppt. of SrC_2O_4.
3. Flame test : intense crimson.

Calcium.

1. Add $CaSO_4$: no ppt.
2. Add H_2SO_4 : a white ppt.
3. Add Ammonium arsenite and NH_4OH : a white ppt. of Calcium arsenite.

   (N.B.—This reagent does not precipitate Sr.)
4. Flame test : brick-red.

Magnesium.

1. Add NH_4OH : a white ppt. of $Mg(OH)_2$. No ppt. is formed if NH_4Cl is present.
2. Add $(NH_4)_2CO_3$: a white ppt. of $Mg(OH)_2MgCO_3$. No ppt. is formed if NH_4Cl is present.
3. Heat the solid on charcoal : a white residue, which when moistened with $Co(NO_3)_2$ and heated again turns pink.

Ammonium.

1. Boil with KOH solution : NH_3 evolved, recognized by its smell and alkaline reaction.
2. Add $PtCl_4$ (and HCl if necessary) : a yellow ppt. of $(NH_4)_2PtCl_6$. This ppt. forms better in the presence of alcohol.
3. Add Nessler's solution : a brown ppt. or coloration, even in very dilute solutions.

Potassium.

1. Add $PtCl_4$ to the solution contained in a watch-glass and stir well : a yellow crystalline ppt. of K_2PtCl_6.
2. Add Hydrogen Sodium Tartrate $NaHC_4H_5O_6$ to a strong solution : a white crystalline ppt. of $KHC_4H_5O_6$ will be formed on shaking vigorously.
3. Add Sodium Cobalti-nitrite and Acetic acid : a yellow crystalline ppt. of $K_2NaCo(NO_2)_6$.

Sodium.

This base forms no ppt. with any reagent, but imparts an intense yellow colour to the flame.

PRELIMINARY TESTS FOR ACIDS.

Experiment	Observation	Inference
1. Add dilute HCl to solid. Afterwards warm gently.	1. Colourless gas evolved, which has no smell and turns Lime-water milky.	CO_2, from a Carbonate.
	2. Colourless gas evolved, which has a fœtid smell and blackens Lead acetate paper.	H_2S, from a Sulphide.
	3. Fuming gas evolved, which has a suffocating smell and turns $K_2Cr_2O_7$ green.	SO_2, from a Sulphite.
	4. Same gas evolved, with a yellow deposit of Sulphur.	Hyposulphite.
2. Heat some of the solid with strong H_2SO_4.	The above acid radicals give off the same gases as before, and also we have :—	
	1. Fuming gas evolved, with acid reaction, and turning $AgNO_3$ milky.	HCl, from a Chloride.
	2. Similar gas evolved, but orange fumes as well, which have a very unpleasant smell.	HBr and Br, from a bromide.
	3. Similar gas evolved, but violet fumes as well.	HI and I, from an Iodide.
	4. Crackling noise in test-tube : yellow gas.	Chlorate.
	5. Reddish fumes, with acid reaction.	NO_2, from a Nitrate.
	6. Two gases evolved, one turns Lime-water milky, the other burns with a blue flame at the mouth of the tube.	CO_2 and CO, from an Oxalate.

Experiment	Observation	Inference
	7. Substance blackens with a smell of burnt sugar: gas given off which turns $K_2Cr_2O_7$ green.	Tartrate.

If none of the above changes occur, the substance may be a Sulphate, Borate, or Phosphate.

To original solution add HCl and $BaCl_2$	A white pt.	Sulphate.
Add to solid strong H_2SO_4 and alcohol. Set it on fire.	The alcohol burns with a green flame.	Borate.
Acidify the original solution with HNO_3 and add Ammonium Molybdate.	A yellow ppt. on warming.	Phosphate.

CONFIRMATORY TESTS FOR ACIDS.

Sulphate.

1. Add Lead acetate: a white ppt. of $PbSO_4$.

2. Heat solid with Na_2CO_3 on charcoal: remove residue and place on a silver coin, then moisten with a drop of water, and a black stain is produced.

Sulphite.

1. Add $BaCl_2$: a white ppt. of $BaSO_3$, readily soluble in HCl.

2. Add a dilute solution of iodine in KI, warm, and the yellow colour disappears.

Hyposulphites (Thiosulphates).

1. Add Lead acetate: a white ppt. of PbS_2O_3, heat, and it turns black, forming PbS.

2. Add a solution of Iodine in KI, it is at once decolorized.

Sulphides.

1. Heat strongly in air on the lid of a porcelain crucible, SO_2 is evolved.

2. Place on a silver coin, and add a drop of dilute HCl; a black stain is produced.

Oxalates.

1. Add $BaCl_2$: a white ppt. of BaC_2O_4, soluble in HCl.

2. Add $AgNO_3$: a white ppt. of $Ag_2C_2O_4$, soluble in HNO_3.

Phosphates.

1. Add NH_4Cl, NH_4OH, and $MgSO_4$: a white ppt. of $MgNH_4PO_4$.

2. Add $AgNO_3$: a yellow ppt. of Ag_3PO_4, soluble in HNO_3 and in NH_4OH.

3. Add $FeCl_3$: a yellow ppt. of $FePO_4$, insoluble in Acetic acid.

Borates.

1. Solutions of Borates in water are alkaline.

2. Add dilute HCl: this solution will turn turmeric paper orange or brown.

Carbonates.

1. Solutions of Carbonates in water are alkaline.

2. If soluble in water, Carbonates give a white ppt. with $BaCl_2$.

Tartrates.

1. Add $BaCl_2$: a white ppt. of $BaC_4H_4O_6$, readily soluble in acids.

2. Add $AgNO_3$: a white ppt. of $Ag_2C_4H_4O_6$; add to this ppt. very dilute NH_4OH, till it is just dissolved, then warm slightly, and a bright mirror of Ag is deposited on the sides of the tube.

Chlorides.

1. Add MnO_2 to solid, then strong H_2SO_4, and warm, when Cl is evolved, recognized by its yellow colour, smell, and bleaching powers.

2. Add $AgNO_3$: a white curdy ppt. of AgCl, insoluble in HNO_3, but soluble in NH_4OH.

Bromides.

1. As in (1) for Chlorides: Br is evolved, recognized by its red colour and smell.

2. Add Chlorine water, then CS_2, and shake well: the CS_2 dissolves the Br and sinks to the bottom of the tube as an orange-coloured layer.

3. Add $AgNO_3$: a yellowish white ppt. of AgBr, insoluble in HNO_3.

Iodides.

1. As in (1) for Chlorides and Bromides: I is evolved, recognized by its violet colour, and turning starch paste blue.

2. As in (2) for Bromides: I is dissolved in the CS_2, and sinks as a violet layer.

3. Add $AgNO_3$: a yellow ppt. of AgI, insoluble in HNO_3.

Nitrates.

1. Add strong H_2SO_4 and metallic Cu: red fumes of NO_2 are given off.

2. Add strong H_2SO_4, cool, and then add slowly a freshly prepared solution of $FeSO_4$: a brown ring appears where the two liquids meet.

3. Add Zn and KOH and warm: the nascent H reduces the nitrate to NH_3 gas, which is given off, and recognized by its smell and alkaline reaction.

Chlorates.

1. Add HCl to solid: Chlorine peroxide (ClO_2), a yellow explosive gas, and Chlorine are evolved.

2. Acidulate with H_2SO_4 and add a little indigo: on warming the indigo loses its colour.

EXAMPLE OF HOW AN ANALYSIS OF A SIMPLE SALT SHOULD BE ENTERED IN THE NOTE-BOOK.

Experiment	Observation	Inference
	The salt is white and crystalline.	Absence of Cu, Fe, Cr, Ni, Co.
Preliminary Tests for the base.		
1. Heated solid in a small dry tube.	Substance did not melt; gave off red fumes which turned blue litmus red; also a gas which rekindled a glowing splinter. No water was given off.	NO_2 and O, from a nitrate. Absence of water of crystallization.
2. Moistened the solid with HCl, and heated on platinum wire.	No flame coloration.	Absence of Ba, Sr, Ca, Na, K, Pb, As, Cu.
3. Heated substance on charcoal before the blow-pipe.	Substance deflagrates. White infusible residue left.	Nitrate or chlorate. Presence of Al, Zn, Mg, Ca, Ba, Sr.
Confirmatory.		
Added two or three drops of $Co(NO_3)_2$ and heated again.	Residue turns blue.	Presence of Al, or a Phosphate.
Group Tests.		
1. Added HCl.	No ppt.	Absence of Ag, Pb, Hg'.
2. Passed H_2S through the same solution.	No ppt.	Absence of Hg''. Cu, Bi, Cd, As, Sb, Sn.

Experiment	Observation	Inference
3 To fresh solution added NH_4Cl and NH_4OH.	White gelatinous ppt.	Al.
Confirmatory Test for base.		
Added KOH.	White ppt. soluble in excess.	Al.
Preliminary tests for Acid.		
1. Added HCl to solid.	Substance dissolves; no gas evolved.	Absence of $''CO_3$, $''SO_3$, $''S$, $''S_2O_3$.
2. Added strong H_2SO_4 to solid and warmed.	Reddish yellow acid fumes evolved.	$'NO_3$.
Confirmatory Tests for Acids.		
1. Added H_2SO_4 and Cu turnings.	Red nitric fumes evolved.	$'NO_3$.
2. Added H_2SO_4, and, when cool, a solution of $FeSO_4$.	Brown ring formed.	$'NO_3$.
3. Added Zn and KOH.	NH_3 evolved, recognized by smell and alkaline reaction.	$'NO_3$.

The Salt is therefore Aluminium Nitrate.

HOW TO RECOGNIZE SOME OF THE MORE COMMON GASES.

Oxygen.

1. Rekindles a glowing splinter.
2. Turns a solution of Pyrogallic acid and Caustic Potash brown.

Hydrogen.

1. Burns with a blue flame and a small explosion, forming water only.

Nitrogen.

1. Has a negative action in all the usual tests applied.

Carbon Dioxide.

1. Puts out a lighted taper.
2. Turns Lime-water milky.

Carbon Monoxide.

1. Burns with a blue flame and no explosion, forming CO_2.

Sulphuretted Hydrogen.

1. Burns with a blue flame having a red edge.
2. Turns paper moistened with Lead acetate black.

Sulphur Dioxide.

1. Fumes in air.
2. Turns paper moistened with Potassium Bichromate solution green.

Hydrochloric Acid.

1. Fumes in air.
2. Turns blue litmus red.
3. Turns Silver nitrate milky.

Ammonia.

1. Turns red litmus blue.
2. Fumes with stopper of Hydrochloric acid bottle.

Nitrous Oxide.

1. Rekindles a glowing splinter.
2. Does not turn Pyrogallic acid and Caustic Potash brown.

Nitric Oxide.

1. Forms red fumes in air.
2. Turns Ferrous Sulphate brown.

Nitrogen Peroxide.

1. Red colour.
2. Turns blue litmus red.

Chlorine.

1. Bleaches litmus.
2. Turns Iodized starch paper blue.

TABLE OF PRESSURES OF AQUEOUS VAPOUR AT DIFFERENT TEMPERATURES

Degrees Centigrade	Pressure in m.m. of Mercury
5·0	6·5
5·5	6·8
6·0	7·0
6·5	7·2
7·0	7·5
7·5	7·8
8·0	8·0
8·5	8·3
9·0	8·6
9·5	8·9
10·0	9·2
10·5	9·5
11·0	9·8
11·5	10·1
12·0	10·5
12·5	10·8
13·0	11·2
13·5	11·5
14·0	11·9
14·5	12·3
15·0	12·7
15·5	13·1
16·0	13·5
16·5	14·0
17·0	14·4
17·5	14·9
18·0	15·4
18·5	15·8
19·0	16·3
19·5	16·9
20·0	17·4

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